THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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THE JOINING OF SOME NICKEL ALLOYS

As-welded joints in Nickel, Monel and Inconel match the tensile properties and corrosion-resistance of annealed parent materials. The tensile strength of as-welded Corronel B is slightly less than that of the annealed parent material, and the full corrosion-resistance adjacent to a weld can be obtained only by heat-treatment after welding. K Monel requires heat-treatment after welding to develop its optimum mechanical properties.

With Nimonic DS and Nimonic 75 as-welded joints will have high-temperature properties similar to those of the annealed parent materials. However, with Nimonic 80, Nimonic 80A, and Nimonic 90 heat-treatment is necessary to develop the optimum high-temperature properties in the parent metal; welded joints may be heat-treated to give similar general properties, but it is not yet possible to develop in the cast weld metal those creep properties that are characteristic of the wrought alloys.

The use of soft-soldered joints is limited by the intended service application. Silver-soldering is frequently used, but it is important to employ the recommended solders on materials in the annealed condition, or stress-cracking may result. High-temperature brazing may be useful for the Nimonic alloys and other heat-resisting materials when welding is impracticable.

Materials to be Joined	Processes which can be used for		Materials to be Used in Various Methods of Joining						
				Inert-Gas Welding					
	Heavy Gauge Material	Light Gauge Material	Metal-Arc Welding A	Tungsten-Arc B	Metal-Arc C	Carbon-Arc Welding D	Oxy-Acetylene Fusion Welding G	Brazing J	Soft Soldering K
NICKEL	A C G (Pipe) H I_2	B D E G H $I I_2$ J K	Nickel flux-coated electrodes e.g., Inco 131	Nickel filler wire Wiggin 61	Nickel filler wire. Wiggin 61, layer-wound spools	Flux-coated nickel filler wire	41 nickel filler wire. Use of Monel welding flux, optional	Silver-copper-zinc brazing alloys, e.g., B.S. 1845, Type 4. Avoid those alloys containing phosphorus. Use proprietary fluxes	Eutectic, 63/37, tin-lead solder, or those quoted in B.S. 219/1949. Resin type flux may be used
MONEL Ni-Cu	A C G (Pipe) H I_2	B D E G H $I I_2$ J K	Monel flux-coated electrodes e.g., Wiggin 130	Monel filler wire Wiggin 60	Monel filler wire. Wiggin 60, layer-wound spools	Flux-coated Monel filler wire	40 Monel filler wire. Monel welding flux	ditto	ditto
K-MONEL Ni-Cu-Al	A I_2	B D E G $I I_2$ J K	Monel flux-coated electrodes e.g., Wiggin 130	'K' Monel filler wire		Flux-coated Monel filler wire	44 K Monel filler wire. Modified Monel welding flux	ditto	ditto
CORRONEL B Ni-Mo-Fe	A	B G	Corronel 'B' flux-coated electrodes	Corronel 'B' filler wire			Corronel 'B' filler wire		
INCONEL	A C H I_2	B D E G	Inconel flux-	Nickel-chrom-	Nickel-chrom-	Flux-coated Inconel filler	Nickel-chromium	Silver-copper-zinc brazing alloys, e.g.,	Eutectic tin-lead solder, or those

			e.g., Wiggin 130				Monel welding flux	ditto	ditto
CORRONEL B Ni-Mo-Fe	A	BG	Corronel 'B' flux-coated electrodes	Corronel 'B' filler wire			Corronel 'B' filler wire		
INCONEL Ni-Cr-Fe	A C H I_2	B D E G I_1 I_2 J K	Inconel flux-coated electrodes e.g., Inco 132	Nickel-chromium filler wire Wiggin N.C.82	Nickel-chromium filler wire Wiggin N.C.82, layer-wound spools	Flux-coated Inconel filler wire	Nickel-chromium filler wire Wiggin N.C. 82. Inconel flux	Silver-copper-zinc brazing alloys, e.g., B.S. 1845, Type 4. Avoid those alloys containing phosphorus. Use proprietary fluxes. Also high temperature brazing using nickel-chromium-boron, nickel-silicon-boron, nickel-chromium manganese and silver-palladium-manganese alloys (S.P.M. 1 and 2)	Eutectic tin-lead solder, or those quoted in B.S. 219/1949. Use vigorous flux e.g., zinc chloride, phosphoric acid or lactic acid types
NIMONIC D.S. Ni-Cr-Fe-Si	A C G	B G J	Nickel-chromium flux-coated electrodes e.g., Inco Rod A, Nicrex 4 or Chromac N	Nickel-chromium filler wire Wiggin N.C.82	Nickel-chromium filler wire Wiggin N.C.82, layer-wound spools		Nickel-chromium filler wire Wiggin N.C.82. Inconel flux	ditto	
NIMONIC 75 Ni-Cr	A C H I_2	B D E G I_1 I_2 J	Inconel (Inco 132) or nickel-chromium flux-coated electrodes e.g., Inco Rod A, Nicrex 4 or Chromac N	Nickel-chromium filler wire Wiggin N.C.82 also Nimonic 75 filler wire	Nickel-chromium filler wire Wiggin N.C.82, layer-wound spools	Flux-coated Nimonic 75 or nickel-chromium filler wire Wiggin N.C. 82	Nickel-chromium filler wire Wiggin N.C.82. Inconel flux	ditto	
NIMONIC 80A Ni-Cr-Ti-Al	A C I_2	B E I_1 I_2 J	Inconel (Inco 132) or nickel-chromium flux-coated electrodes e.g., Inco Rod A, Nicrex 4 or Chromac N	Nickel-chromium filler wire Wiggin N.C.82 also Nimonic 80A or Nimonic 90 filler wire	Nickel-chromium filler wire Wiggin N.C.82, layer-wound spools			ditto	
NIMONIC 90 Ni-Co-Cr-Ti-Al	A C I_2	B E I_1 I_2 J	Inconel (Inco 132) or nickel-chromium flux-coated electrodes e.g., Inco Rod A, Nicrex 4 or Chromac N	Nickel-chromium filler wire Wiggin N.C.82 also Nimonic 90 filler wire	Nickel-chromium filler wire Wiggin N.C.82, layer-wound spools			ditto	
AUSTENITIC CHROMIUM-NICKEL STAINLESS STEELS	A C F H I_2	B D E F G I_1 I_2 J K	Several proprietary types of electrode based upon appropriate composition	Matching filler wires	Matching filler wires	Flux-coated stainless steel wire	Matching filler wires. Appropriate flux	ditto	Eutectic tin-lead solder, or those quoted in B.S. 219/1949. Special, vigorous fluxes required

A Metal-Arc Welding
B Inert-Gas Tungsten-Arc Welding
C Inert-Gas Metal-Arc Welding
D Carbon-Arc Welding
E Atomic-Hydrogen Welding
F Submerged-Arc Welding
G Oxy-Acetylene Fusion Welding
H Oxy-Acetylene Pressure Welding
I Resistance Welding Spot and Seam
I_2 Flash-Butt Welding
J Brazing
K Soft Soldering

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Reference Data on Metals and Alloys

C. J. SMITHELLS: 'Metals Reference Book.'
2nd, revised and enlarged, edition.
Published, in two volumes, by *Butterworths Scientific Publications, London*, 1955; 967 pp. + index.
Price £8 8s. 0d.

The aim of the first edition of this reference book, published in 1949, was to provide a summary of critically correlated data relating to metallurgy and metal physics. The material contained in it was prepared by some 40 individual specialists in the respective fields covered, and the editor-in-chief was assisted by seven associate editors well known in the metallurgical field. This plan has been adhered to in the new edition, with the further assistance of contributors covering aspects not dealt with at the earlier stage, e.g., elastic properties and damping capacity, physical properties of molten salts, and friction. In addition to very extensive revision of individual values, in the light of recent research, some sections of the book have been entirely rewritten, including those on crystal chemistry, diffusion in metals, thermochemical data, and welding. A further change is the amplification of *bibliographies*, which are given at the end of each section.

The wide scope of the new edition will be obvious from the following skeleton list of subjects covered:—

Volume I—Introductory tables; general physical and chemical constants
- Atomic nuclei and their properties
- Line spectra of the elements
- X-Ray crystallography
- Crystal chemistry
- Geochemistry
- Metallography
- Equilibrium diagrams (binary, ternary and quaternary)

Volume II—Gas-metal systems
- Diffusion in metals
- Elastic properties and damping capacity
- Thermochemical data
- Physical properties of molten salts
- General physical properties of metals, alloys and steels
- Thermoelectric properties and temperature measurement
- Radiating properties of metals
- Thermionic, photoelectric and secondary emission
- Electrical properties
- Steels and alloys having special magnetic properties
- Mechanical testing
- Mechanical properties of metals, alloys and steels
- Hard metals
- Deep-drawing properties
- Lubricants
- Casting alloys and foundry data
- Refractories
- Fuels
- Carbon and graphite electrodes
- Controlled atmospheres for heat-treatment
- Corrosion
- Electroplating and metal finishing
- Welding, soldering and brazing
- Miscellaneous data.

As in the first edition, the information is given, wherever possible, in the form of tables, graphs and other forms of figure.

The demand which has called for issue of a second edition will ensure a welcome to this unusually comprehensive reference book.

Nickel-Alloy and Other Thermocouples: Reference Data

H. SHENKER *et al.*: 'Reference Tables for Thermocouples.' *Nat. Bur. Standards Circular* 561, 1955; 84 pp.
Published by *U.S. Govt. Printing Office, Washington 25, D.C.* Price 50 c.

One of the major functions of the National Bureau of Standards is to compile scientific and technical data which have broad and useful application in science and industry. In many cases the programmes are carried out in co-operation with scientific and industrial organizations which themselves require accurate basic information in specific fields. One project of this type has been concerned with the preparation of up-to-date temperature/electromotive-force tables for thermocouples, the testing of commercial couples, the preparation of purchase specifications for thermocouple wire, and the precise calibration of the scales of instruments.

The reference tables contained in this Circular have been prepared to make generally available, in composite and convenient form, data previously issued in Bureau publications which are now out of print, and to meet the continuing need of industry and scientific investigators for reliable data on currently accepted values.

The types of thermocouples for which tables are given (both Celsius and Fahrenheit) are shown on p. 175.

Platinum *vs.* Platinum—10% rhodium
Platinum *vs.* Platinum—13% rhodium
Chromel–Alumel
Iron–Constantan
Copper–Constantan
Chromel–Constantan

The tables are based on the absolute electrical unit and the International Temperature Scale of 1948.

Thermal Conductivities of Metallic Materials at Sub-Zero Temperatures

R. W. POWELL: 'The Thermal Conductivities of Metals Below Room Temperature.' *Bull. Inst. International du Froid, Annexe*, 1954-2, pp. 111-18.

Although there has been considerable research on low-temperature thermal conductivities of metals and alloys, and several comprehensive summaries of information have been published, most of the information available relates to work done at cryogenic temperatures, well below atmospheric level. In the review now published an attempt has been made to relate the measurements made at cryogenic temperatures with data for normal and higher temperatures. For certain practical applications, information is required on conductivity in the immediate sub-atmospheric temperature region, and in this connexion it has become evident that data for the temperature range between liquid oxygen and melting ice are relatively sparse.

In order to derive some indication of the trends within that range, the author has plotted a selection of data obtained from eighteen different sources, covering irons of varying purity, carbon steels, low- and medium-alloy steels, and high-alloy steels. In the first place thermal conductivities of materials of these types are shown as a function of temperature, 0°-1000°C., and the survey is later extended to include sub-atmospheric data correlated, where possible, with elevated-temperature values. The range covered is 1° to 1273°K. (−274° to +1000°C.). Correlation of the data from the two forms of presentation reveals that the variation of thermal conductivity with degree of alloying and with temperature follows a sufficiently definite pattern to make possible the prediction of approximate values for temperature regions for which experimental data are lacking. The values taken into account in this analysis relate almost entirely to steels in the annealed condition: little information is available for these materials in any other conditions. The author points out, however, by reference to determinations which have been made on non-ferrous materials as well as the few investigations on steels, that cold work, quenching, and tempering have a pronounced influence on thermal conductivity, and that future investigations of this property should be made on materials in various physical conditions.

See also—

R. W. POWELL: 'Thermal Conductivities of Metals and Alloys at Sub-Zero Temperatures',
ibid., Annexe, 1955-2, pp. 115-35.

This paper, complementary to the previous one, is concerned with non-ferrous metals and alloys. The author points out the very wide scope of this field, and emphasizes that the review is not to be considered exhaustive. The aim has rather been (as in the case of the data on ferrous materials reported earlier) to present a general picture, based on authentic data on typical materials. Particular attention is given to relating low-temperature thermal conductivities, where marked dependence on purity and mechanical condition is observed, to values at higher temperatures, where such variables have a reduced effect. The data are considered in four broad groupings: (1) copper and copper-base alloys; (2) metals and alloys for which there are experimental data for the temperature range immediately below normal; (3) metals for which data in that region are not available; (4) graphite and carbons. Some of the general conclusions drawn from experimental and extrapolated values are given below:—

For materials in category (2) experimentally determined values for temperatures between those of liquid oxygen and of ice agree in general with those found for higher and lower regions. For metals in group (3) values for the region immediately below normal were extrapolated, to give values for −100° and −200°C. At relatively low temperatures the thermal-conductivity values for pure metals go through a maximum and subsequently decrease linearly to zero at 0°K. (−273°C.). As the purity of the metal increases thermal conductivity rises, and the temperature at which the characteristic sub-zero maximum occurs falls.

The report also includes suggestions for further research on thermal conductivity, and calls attention to errors in values which have been taken as standard for iridium and rhodium.

Cladding of Metals by Vacuum Brazing

K. ROSE: 'New Clad Metals made by Vacuum Brazing.' *Materials and Methods*, 1955, vol. 42, July, pp. 100-2.

This article gives a general outline of a new method of production which is expected to widen the scope of application of clad materials. According to this process, developed by the Chicago Bridge and Iron Company, the two metallic materials which are to be bonded are placed in a furnace with a thin foil of brazing alloy between them. As the brazing alloy melts a vacuum is drawn between the two surfaces and the two materials are forced together by external atmospheric pressure. The vacuum serves two purposes:—

(1) it draws away any gases which may be liberated at the interface and might oxidize the bonding surfaces. By preventing oxidation, the use of the vacuum method eliminates the need for introduction of a special atmosphere; it also prevents contamination by brittle particles of flux which are often entrapped in conventional flux-brazing of large continuous surfaces,

(2) it provides uniform bonding pressure, which is maintained throughout the brazing process, and only a thin layer of brazing alloy is required,

which permits a high proportion of it to alloy with the steel base, giving joints of high shear strength in relation to the duration of the brazing cycle involved.

Penetration of copper varies with different metals and alloys, and the degree of penetration must be controlled by suitable placing of the material, thickness of the brazing layer, and duration of the brazing cycle. In stainless-clad steels copper or copper-alloy brazing material not only bonds, but also serves as a barrier to carbon migration from the base to the cladding material. In rolled stainless-clad steel the same function is performed by a layer of nickel, plated onto the stainless steel prior to vacuum-brazing.

The writer emphasizes the advantages of the new process, e.g., it requires no heavy rolling equipment, soaking pits, etc., and the size of material which can be handled is limited only by the capacity of the brazing furnaces available.

Composite materials made by the process described have been found to show properties well within the limits for clad materials laid down by current specifications, and are reported to have given satisfactory service for several years as materials of construction for pressure vessels subjected to process temperatures up to 900°F. (480°C.). The most widely used types are those having a carbon-steel backing with nickel, nickel-alloy or stainless-steel cladding, and for special orders cladding of carbon steel with Hastelloy (nickel-molybdenum-base) alloys has also been successfully carried out. Silver-clad stainless steel and cladding of both plain and stainless steels with titanium are among other applications for which the vacuum-braze process is suitable. The products made by this method are designated 'Hortonclad.'

Vibrational Treatment of Austenitic Steel Castings during Solidification

See abstract on p. 195.

Metallic Materials for Nuclear-Fuelled Power Plants

D. O. LEESER: 'Evaluating Basic Materials for Nuclear-Fuelled Power Plants.' *Mechanical Engineering*, 1955, vol. 77, June, pp. 501–4.

This review is prefaced by a plan showing the arrangement for a steam-power plant utilizing heat energy from a nuclear reactor. Properties required in materials for the respective parts of the plant are then considered, in the light of their behaviour in the conditions operative in the various components. Individual sections of the review deal with core and control materials, materials used in auxiliary systems of the plant, and heat-transfer materials (the latter group with special reference to corrosion aspects). A number of useful tables are given, showing some basic data which can indicate the suitability of specific metals or alloys for applications in nuclear-power plant. These include information on slow neutron absorption by various metals and by stainless steel, and the influence of irradiation on the hardness and the tensile properties of structural materials used in reactors. The materials for which such data are given include stainless steels and nickel.

Materials of Construction in Ore-Treating Plants

See abstract on p. 195.

NICKEL

Sherritt Gordon Nickel Mines: Historical Survey of Development

A series of papers on the Sherritt Gordon Lynn Lake Project appears in *Canadian Mining and Metallurgical Bulletin* for June and July 1955. The scope of the individual papers, and their location, are indicated below.

E. L. BROWN: 'Notes on Discovery and Financing.' *Canad. Mining and Metallurgical Bull.*, 1955, vol. 48, June, pp. 335–9.

This article describes discovery, in 1941, of sulphide ores containing about 1·5 per cent. of nickel, with rather less than 1 per cent. of copper, and traces the various steps leading up to exploitation of the orebody, and extraction of nickel by the ammonia-leaching process worked out by Professor Forward in co-operation with Sherridon chemists.

Financial considerations involved in working these ores are discussed, and development to the end of 1954 is outlined. Rather less than nine years after the first discovery of the orebody in this region the initial shipment of nickel left the Fort Saskatchewan plant, on August 7th, 1954. Lynn Lake has now become Canada's second largest nickel-producing area, and is likely to make an increasingly important contribution to the world demand for nickel.

G. D. RUTTAN: 'Geology of Lynn Lake', *ibid.*, pp. 339–48.

A detailed historical record of the nature and formation of the minerals found, with notes on the metal values of the respective components.

J. R. MUTER: 'Mining at Lynn Lake', *ibid.*, July, pp. 385–9.

The two orebodies already in operation by the Sherritt Gordon Mines are some two miles apart and are mined as separate units. The ore from one of these is of higher grade than that produced in the other area, and the two are mixed, to provide as uniform a feed as possible for the grinding mills. This article describes initial development, and mining methods currently used. Refinements in operation now being introduced are expected to reduce costs.

R. T. DRAKE: 'Milling at Lynn Lake', *ibid.*, pp. 390–5.

Crushing and milling operations are described in detail, with flow sheet, to the stage at which the nickel concentrate is sent to the Company's refinery at Fort Saskatchewan and the copper concentrate to Noranda, Quebec. Details are given of methods used for routine assaying for nickel, copper, and insoluble residue (during each shift and prior to dispatch of the concentrate). Procedure used for cobalt assay is also noted.

S. NASHNER: 'Refining at Fort Saskatchewan', *ibid.*, pp. 396–410.

Previous papers by individuals associated with the development of the ammonia-leaching process used at the Sherritt Gordon refinery have already discussed the reasons leading to selection of this process for treatment of the Lynn Lake ores, and have described the essential features of the process: see, for example, *Trans. Canad. Inst. Min. Met.*, 1948, vol. 51, pp. 181–6; 1953, vol. 56, pp. 373–80; *Bull. Inst. Metals*, 1954, vol. 2, pp. 113–16.

The present paper is concerned solely with a description of the refinery in commercial operation. The layout is illustrated, a description is given of the various unit operations and the major items of equipment used in them, and some comparison is made of process results obtained at the refinery with those predicted by and obtained in laboratory-scale work. It has been found that, in general, the chemical-process results in large-scale operation follow closely those obtained in the pilot plant, and, with regard to materials selected for the plant, it is stated that corrosion rates calculated on the basis of experience in the pilot plant have been confirmed within the limits of the relatively short operating experience in the full-scale installation.

Nickel-containing Ores of Cuba. Extraction Methods: Prospects of Development

W. D. MCMILLAN and H. W. DAVIS: 'Nickel-Cobalt Resources of Cuba.'
U.S. Bur. Mines, Report of Investigations 5099, Feb., 1955; 86 pp.

A review of the world reserves of nickel-containing ores, with notes on the individual types which are found in various areas in North America, South America, Europe, Africa, Asia and Oceania. This information is introductory to a detailed report on the significance of nickeliferous ore reserves in Cuba, which form the world's largest potential source of nickel and cobalt, and an important potential source of iron and chromium. The history of wartime development of the lateritic Cuban ores is recorded, and methods which have been used for extraction of nickel from them are described, with flow sheets.

Following a non-productive period (1947–1952), further exploitation of the Cuban reserves was again considered, and this report traces the results obtained to the end of 1954, on the basis of which critical consideration is given to the economic feasibility of further development.

Ammonia Leaching of Nickel-containing Ores: Summary of Processes Used

M. H. CARON: 'Ammonia Leaching of Nickel Sulphide Ores: Half Century Review.'
Trans. Instn. Mining and Metallurgy, 1955, vol. 64, Aug., pp. 611–16.
Abstract in *Chemical Trade Jnl.*, 1955, vol. 137, Aug. 12, pp. 413–14.

The review covers the salient points relating to processes proposed for ammonia leaching of nickel sulphide ores, and notes developments since the patenting, in 1903, of a process invented by Malzac, up to the present day, when two main methods are in large-scale operation.

Processes for the treatment of certain types of nickel-bearing ore were patented by the author in 1923, and formed the basis of methods used, as a wartime measure, for extraction of nickel from the low-grade ores at Nicaro, Cuba.* Later a modified process, evolved from the Caron procedure, was worked out jointly by Professor Forward, of the University of British Columbia, Vancouver, and The International Nickel Company. Nickel-bearing sulphide ores, of the extremely refractory pyrrhotite type, are the initial material, and from this, by judicious suspension roasting and a controlled selective reduction of the calcine, the nickel is extracted in a counter-current leaching system, using ammonia-ammonium carbonate solution. From the solutions so produced nickel is recovered by distillation as basic nickel carbonate, and ammonia and carbon dioxide are returned for re-use.

The other ammonia-leaching process which has been developed to a commercial scale is based on the original Malzac procedure: it is being operated, after initial research by Forward and his colleagues, by Sherritt Gordon Mines, Ltd. A salient feature of the Malzac method is fine grinding of the ores, and their direct treatment with strong ammonia liquor and air, to oxidize sulphide sulphur and to form soluble ammonia complexes of nickel. This process involves aeration at atmospheric or higher pressures.

The author of this review makes some comparison of the relative advantages and disadvantages of the two methods. It is pointed out that the pressure-ammonia process has the advantage of direct production of metals, elimination of roasting and reduction, and the recovery of sulphur as ammonium sulphate. It suffers, however, from the inherent difficulties involved in pressure operation, and from the considerable heat released by oxidation of the sulphides. A further limitation is that although the process can be used for pentlandite, it is unsuitable for treatment of such low-grade concentrates as nickeliferous pyrrhotite. In addition, recovery of sulphur is restricted to production of ammonium sulphate.

The International Nickel Company's process has the advantage of continuous operation, and ammonia leaching is effected at normal atmospheric pressure, facilitating bulk treatment of low-grade ores. It is suitable for treatment of both pentlandite and pyrrhotite, and it offers wide flexibility in choice of methods for recovering sulphur. In addition, the roasting stage involved in the process can produce more than sufficient steam for process purposes. The INCO process has the comparative disadvantage of calling for substantial fuel consumption in the reducing stage, and the nickel is produced as oxide, not metal.

The review contains annotations on the major literature concerned with the principles and development of ammonia leaching of ores: references to original technical and patent publications are given in a selected *bibliography*.

**See also preceding abstract.*

Nickel-Sulphur and Steel-Sulphur Reactions

A. DRAVNIEKS: 'Kinetics of Nickel-Sulphur and Steel-Sulphur Reactions.' *Jnl. Electrochemical Soc.*, 1955, vol. 102, Aug., pp. 435-9.

Although the susceptibility of nickel and some nickel alloys to sulphur is well known, there are few data on the kinetics of formation of nickel sulphide, especially in moderately elevated temperature ranges. The aim of the work reported in this paper was to provide such data, and to compare them with observations on iron-sulphide formation which had previously been made (*Ind. Engg. Chem.*, 1951, vol. 43, p. 2897).

Molten sulphur was used as the medium, and the experiments were made by two methods:—

1. triplicate samples of commercial nickel (45 mm. $\times$8 mm.$\times$0·62 mm.) were immersed in molten sulphur at temperatures between 205° and 445°C., for various periods. On removal, the specimens were quenched in water, and weight-loss was determined after mechanical removal of the sulphide scale. (A short pickling in 10 per cent. sulphuric acid facilitated scale removal.) Average values from the triplicate samples were used for compilation of reaction-time curves.
2. Electrical conductivity of commercial nickel strips immersed in molten sulphur was followed as a function of time. Technique of interpretation of these measurements was described in earlier papers (*Ind. Engg. Chem.*, 1951, vol. 43, p. 2897; *Corrosion*, 1954, vol. 10, p. 224).

The observations made, considered in the light of earlier work on iron-sulphide formation and of other investigations on nickel-sulphur reactions, lead to the following conclusions:—

Reaction of nickel with molten sulphur between 205° and 445°C. follows the parabolic law. Up to 260°C. the millerite form of NiS was the only sulphide detectable in the scale. At 340°C. and above the high-temperature B8 form of NiS was the only sulphide present. The activation energy below about 300°C. is in excess of 50 kcal.: at that temperature it changes abruptly and at higher temperatures it is of the order of 20 kcal. (This value compares with an activation energy of 41 kcal. for the iron-sulphur reaction over the entire temperature range studied, where only one FeS form is known.) Upon exhaustive reaction of the corresponding mixtures of nickel and sulphur, Ni_3S_2, Ni_6S_5, two forms of NiS, and NiS_2 were found to form, but there was no indication of formation of Ni_7S_6 or of Ni_3S_4, and in view of the author's own work and that of other investigators it is considered doubtful whether such compounds exist. Formation of NiS_2 was several thousand times slower than that of NiS. The latter sulphide formed continuously.

Sulphur Activity in Liquid Nickel

See abstract on p. 186.

Effect of Sulphur in Nickel

I. PFEIFFER: 'The Behaviour of Sulphur in Nickel, and Diffusion Phenomena.'
Zeitsch. f. Metallkunde, 1955, vol. 46, July, pp. 516-20.

The author refers to classic investigations which have indicated that the cause of embrittlement in nickel containing sulphur is the formation of a network of nickel sulphide. It is pointed out that Masing and Koch studied the influence of sulphur contamination down to amounts of the order of 0·02 per cent., but that in commercially pure nickel the sulphur content is well below this level, e.g., about 0·003-0·004 per cent., and it is with percentages of this lower order that the present paper is mainly concerned.

Metal prepared by intentional contamination of very pure nickel with 0·006 per cent. of sulphur, during melting, was compared with commercial nickel containing 0·003 per cent. of sulphur, and it was found that whereas the latter was brittle, the contaminated nickel of high initial purity showed no brittleness and that the structure was finer-grained and showed no balling up of nickel sulphide at the grain boundaries. It was therefore concluded that sulphur in such small amounts is not responsible for embrittlement, which must be attributed to other impurities present. Further studies, in which nickel of high purity was contaminated with oxides, produced a brittle material, leading to the conclusion that oxides may be regarded as a harmful factor. An addition of carbon to the nickel melt inhibited embrittlement: this is believed to result from reduction of the oxides present in the molten metal, not from removal of sulphur, since reaction between sulphur and carbon in the melt is thermodynamically impossible.

In view of these findings, a fundamental investigation was made of the mechanism of diffusion of sulphur in nickel, and of the rate of diffusion. Photomicrographic examination indicated that with a content of 0·006 per cent. the sulphur is homogeneously distributed in the nickel, with no preferential balling up at the crystal boundaries, confirming the observation that no brittleness was found in the pure nickel containing that amount of sulphur. Nickel annealed at 1200°C. showed clearly marked concentration of sulphur in individual grains; this is considered by the present author to be due to preferential diffusion into highly stressed crystals.

The diffusion coefficient of sulphur in nickel was determined at 1000°, 1100° and 1200°C., using nickel into which radioactive sulphur was introduced. The high activation energy found in this region, 90,000 cal./mole, leads to the conclusion that the mechanism is one of pure lattice diffusion.

Reference Data on Metals and Alloys

See abstract on p. 174.

Thermal Conductivities of Metallic Materials at Sub-Zero Temperatures

See abstract on p. 175.

Oxidation of Nickel: Initial Stages

U. M. MARTIUS: 'The Initial Oxidation of Nickel.' *Canad. Jnl. of Physics*, 1955, vol. 33, Aug., pp. 466–72.

Attention is called to the sparsity of data available on the mechanism of nucleation of an oxide layer on the surface of a metal crystal, i.e., the initial stages of oxidation. The work now reported was carried out on cold-rolled nickel sheet of high purity, prepared by mechanical polishing, annealing for two hours at 1100°C., and final electrolytic polishing. The specimens were then heated to 1100°C. under a stream of hydrogen containing a trace of water vapour, and were kept at that temperature for times varying from 10 minutes to 2 hours, followed by slow cooling, under hydrogen, to room temperature. Such conditions yield material at the initial stage of oxidation: the surface is not yet covered with a continuous film of oxide (no interference colours are visible), but small individual crystallites (or nuclei) have formed on it.

Each grain of nickel, depending on its crystallographic orientation, shows variation from any other, in the number, size and orientation of the oxide crystallites on its surface. Photomicrographs in this report illustrate typical structures observed. Particular attention is directed to oxidation phenomena observed at grain boundaries: it is noted that (*a*) some grain boundaries show a string of oxide crystallites, aligned along the grain boundaries rather than following the shape and orientation of the oxide crystallites on the surface of the grains on either side of the boundary: (*b*) some boundaries show oxide crystallites of an orientation and shape similar to those of the oxide crystallites on one of the neighbouring grains, and (*c*) certain grain boundaries, especially twin boundaries, exhibit no oxidation pattern of their own. (Examples of all three types of formation are given.)

In interpretation of the experimental findings, the author makes the following comments: 'It is known that the rate of oxidation is determined by self-diffusion. At the points on the nickel surface at which visible oxide crystallites are found during initial oxidation the 'local' rate of diffusion of nickel must have been high. The surface points at which the oxide crystallites begin to form and continue to grow are definitely related to the crystallography of the underlying metal surface. These 'growth sites' will be positions at which nickel can diffuse preferentially, whether on account of the geometry of the lattice, or on account of existing lattice imperfections. The observations at grain boundaries strongly stress the rôle of certain crystal imperfections, probably screw dislocations, in the process of oxidation of metals. The fact that twin boundaries do not show oxide patterns of their own is another supporting indication.'

Adsorption of Gases on Nickel Oxides

S. J. TEICHNER and J. A. MORRISON: 'The Adsorption of Gases on Nickel Oxides.' *Trans. Faraday Soc.*, 1955, vol. 51, July, pp. 961–6.

The experiments described in this paper were made in connexion with the use of nickel oxide as an oxidation-reduction catalyst: an attempt was made to elucidate the adsorption of simple electron-donor and electron-acceptor gases.

The adsorption of several simple gases, on stoichiometric and non-stoichiometric (excess of oxygen) nickel oxides was studied, using a gravimetric method. The oxides were prepared by thermal decomposition of pure nickel hydroxide. Special attention was given to the chemisorption of carbon monoxide and oxygen, and to the interaction of these gases on the surface of the oxides at room temperature. In contrast to findings earlier reported for films of nickel oxide on nickel metal (*ibid.*, 1954, vol. 50, p. 501–10), the authors conclude that carbon monoxide is adsorbed on these oxides to a much greater extent than is oxygen.

The interaction product of carbon monoxide and oxygen on the surfaces is stable at room temperature, but decomposes completely at 200°C.

Heat of Formation of Nickel Carbonyl

K. W. SYKES and S. C. TOWNSEND: 'The Heat of Formation of Nickel Carbonyl.' *Jnl. Chemical Soc.*, 1955, July, pp. 2528–9.

A survey of values recorded in the literature for the heat of formation of nickel carbonyl (*ibid.*, Jan., p. 100) contained also unpublished results obtained in the laboratories of The Mond Nickel Company, Ltd., Clydach. This note gives details of the experimental work on which the latter values were based.

Preparation of Nickel Catalysts from Mixed Oxalates

W. LANGENBECK, H. DREYER and D. NEHRING: 'A Highly Active Nickel Hydrogenation Catalyst.' *Naturwissenschaften*, 1954, vol. 41, p. 332.

Catalysts claimed to be highly active are made by decomposition of mixed nickel and magnesium oxalates. A solution of 25 g. $Ni(NO_3)_2.6H_2O$ and 225 g. $Mg(NO_3)_2.6H_2O$ in 600 c.c. of water is precipitated at 50°C. with a solution of 122 g. $(CO_2H)_2.2H_2O$ in 500 c.c. of water. After heating for four hours at 350°C. in a current of hydrogen a black pyrophoric powder is formed, containing nickel and undecomposed magnesium oxalate. This substance hydrogenates 5 c.c. of cyclohexene in 35 c.c. of benzene (at room temperature and atmospheric pressure) in 15 minutes, one-fifth the time required with a Raney nickel catalyst. It is considered that the rigid skeleton of magnesium oxalate is a favourable feature.

Improved Raney Nickel Catalysts

'Raney Nickel Catalyst: Stabilized Form.' *Chemical Age*, 1955, vol. 73, Sept. 24, p.674.

The practical value of Raney nickel catalysts in bringing about numerous reductions with hydrogen, under moderate conditions of temperature and pressure, is now well established. The catalyst is prepared by treatment of a powdered alloy of aluminium and nickel with hot sodium-hydroxide solution, by which the aluminium is dissolved and the nickel is left as a heavy, finely divided powder, which is stored under water or alcohol. Catalytic activity tends to fall during storage, and it has previously

been necessary, in order to ensure optimum behaviour, for the full cycle of preparation to be carried out each time catalyst material was required. Until recently it has not been possible for manufacturers to supply ready-made catalyst, due to the dangers of handling (since it is pyrophoric when dry), and to the impermanence of the catalytic activity under conditions involving exposure to the air. These difficulties have now been overcome by the development, by British Drug Houses, Ltd., of a stable non-pyrophoric preparation from which the fully active catalyst can be regenerated by simple washing with alcohol.

Calcium-Nickel Phosphate Catalysts in Butadiene Manufacture

'Dehydrogenation of Butenes with a Calcium-Nickel Phosphate Catalyst.' *Reprints issued by International Nickel Co., Inc.*; 16 pp.

This composite publication contains reprints of the following papers:—

E. C. BRITTON, A. J. DIETZLER and C. R. NODDINGS: 'A Calcium-Nickel Phosphate Dehydrogenation Catalyst' (from *Industrial and Engineering Chemistry*, 1951, vol. 43, Dec., pp. 2871–4).

This paper describes the initial work done, in Dow Chemical Company's research laboratories, in development of an effective catalyst for dehydrogenation of *n*-butenes to 1,3-butadiene, using superheated steam as heating medium and diluent. The catalyst consists of calcium-nickel phosphate, promoted with chromium oxide. The composition of the basis corresponds approximately to the formula $Ca_8Ni(PO_4)_6$. The preparation of the catalyst, its physical properties, use, and performance in laboratory units, are discussed. Selectivity did not change in $3\frac{1}{2}$ months of continuous operation in the laboratory tests, and remained constant at all conversion levels except at temperatures at which it was affected by thermal cracking. The authors draw particular attention to the high *n*-butene utilization, at high conversions, which are realized with the calcium-nickel phosphate catalyst: it is stated that in commercial trials such catalysts have significantly increased plant capacity for 1,3-butadiene, which is an important rubber intermediate.

P. M. REILLY: 'The Commercial Development of Dow Type B Butylene Dehydrogenation Catalyst.' (from *Chemistry in Canada*, 1953, vol. 5, Mar., pp.25–9).

This paper describes the commercial introduction of the catalyst referred to above. The information given is based specifically on its use by Polymer Corporation, Ontario, in a synthetic-rubber producing plant.

C. M. FINIGAN, H. V. FULLERTON and G. W. TAYLOR: 'Carbonyl Compounds as By-Products in Commercial Dehydrogenation of Butenes' (from *Industrial and Engineering Chemistry*, 1953, vol. 45, June, pp. 1356–9).

Subsequent to the use, by the Polymer Corporation, of the calcium-nickel phosphate catalyst, it was found that approximately 0·75 per cent. of the hydrocarbon feed was being converted to water-soluble carbonyl-type compounds. Investigation of this side reaction was considered desirable, in view of the substantial loss of hydrocarbon involved, and of the potential value of the by-products formed. As a background to their own report, the authors of this paper refer to two detailed descriptions of the commercial dehydrogenation process. They then give experimental data furnishing information on total formation of carbonyl during the life of the catalyst and the quantitative distribution of the individual carbonyl constituents formed. They postulate a mechanism to explain the formation of carbonyl compounds during the dehydrogenation reaction.

See also—

C. R. NODDINGS, S. B. HEATH and J. W. COREY: 'Conservation of Butylenes in Butadiene Manufacture.' *Industrial and Engineering Chemistry*, 1955, vol. 47, July, pp. 1373–6.

This article describes the development, production and performance of the promoted calcium-nickel phosphate catalyst. The full story of the problems encountered in the early stages, methods of test employed, and economic factors involved in adoption of the catalyst are critically reviewed, on the basis of experience in the plant of Dow Chemical Company (who have for many years been interested in production of synthetic rubber), of information given by several operators of butadiene plants, and of data supplied by the Reconstruction Finance Corporation. Outstanding problems, arising from premature ageing of the catalyst, are discussed in relation to the economics of its adoption on a larger scale.

Nickel Catalysts in Re-forming of Firedamp for Production of Town Gas

J. G. POPE and B. RICHARDSON: 'Firedamp Re-forming.' *Gas World*, 1954, vol. 190, Nov. 6, pp. 1137–40.

The paper gives an account of experiments made, at coal pits in the Whitehaven area, to re-form the methane drained from the pits, in order to yield a mixture conforming to the combustion characteristics and calorific value of town's gas. Re-forming is effected by passing a mixture of methane and steam through a heated catalyst bed, to promote the reaction expressed by the following equation:—

$$CH_4 + H_2O = CO + 3H_2$$

The catalyst found to be most suitable is firebrick broken to pea size and impregnated with 2–3 per cent. of nickel. Method of preparation is described. Such catalysts have been found to be as satisfactory as those of higher nickel content. Tests are in hand to determine the feasibility of carrying out the reactions in tubes made from 60–20–20 nickel-chromium-iron or other heat-resisting material. Prospects for development of the scheme are discussed, from both the technical and the economic aspects.

Nickel in Ultrasonic Equipment for Protection of Ships' Hulls from Barnacles

'Ships' Hulls Protection. Ultrasonic Vibrations make Barnacles Uncomfortable.' *Engineering*, 1955, vol. 180, Sept. 23, p. 416: see also *The Times*, 1955, Sept. 6, p. 7.

Ultrasonic vibrations are being successfully used to discourage the settlement of barnacles on the hulls of ships and to inhibit the growth of barnacles which have already settled. The method of producing the vibrations is to solder a transducer onto a plate, weld the plate to the inside of the ship's hull, and induce vibrations of 23–27 kc/s. into the hull plates through the transducer. These vibrations, which are sonic rather than mechanical, make the hull an uncomfortable place for barnacles to settle, but the effect is psychological only, and no physical injury to life is involved.

This article makes a record of some cases in which the method has been adopted for treatment of the hulls of ocean-going ships, and points out the advantages in saving of maintenance time and expense which can be so achieved. It has been ascertained that the ultrasonic vibrations do not adversely affect personnel or instruments in the ships.

In the equipment described, nickel is used as the ultrasonic medium.

Metallic Materials for Nuclear-Fuelled Power Plants

See abstract on p. 176.

Influence of Nickel on Plant Culture

J. G. HUNTER: 'Nickel Toxicity in a Southern Rhodesian Soil.' *South African Jnl. of Science*, 1954, vol. 51, Dec., pp. 133–4.

Infertility associated with soils derived from serpentine and related rocks has been the subject of investigation in many parts of the world. The author briefly refers to some of the relevant literature, calling particular attention to experimental work carried out by himself and Vergnano, in which toxicity of certain trace elements was studied by growing oat plants, under standard conditions, in nutrient solutions in which separate elements were present at various concentrations, and in soils containing various percentages of individual elements. The tests indicated that in soils derived from serpentine rocks nickel was the element responsible for toxicity. Later experiments, reported in the present paper, confirmed the finding that excess of nickel, such as is likely to occur in such soils, is detrimental to growth of oats, and demonstrated that tobacco plants are also adversely affected.

Photometric Determination of Nickel in Steel

K. SPECKER and H. HARTKAMP: 'Photometric Determination of Nickel, Aluminium and Manganese in Crude Steel.' *Zeitsch. anal. Chemie.*, 1955, vol. 145, pp. 260–5.

The abstract below is quoted from *Chemical Abstracts*, 1955, vol. 49, pp. 8737–8.

'Treat 0·1–0·01 g. of sample with aqua regia, evaporate to dryness with NaCl, and repeat the evaporation with strong HCl to remove all nitrate. Transfer to a 200-ml. graduated funnel, add 10 ml. concentrated HCl, and dilute to 30 ml. While cooling add 30 ml. Et_2O and 30 ml. tetrahydrofuran, followed by 15 ml. NH_4CNS reagent. Shake and separate the layers. Add 10 ml. more of NH_4CNS and 20-ml. portions of Et_2O and tetrahydrofuran and finally shake with 20 ml. Et_2O or $CHCl_3$. Discard all the organic phases. To the aqueous phase add 2–3 ml. concentrated NH_4OH, 50 ml. buffer solution, pH 5·5–6·0, and 5 ml. dimethylglyoxime reagent. Extract out the nickel precipitate with 10 ml. $CHCl_3$.

Treat with 10 ml. water and 5 ml. aqueous bromine and read the extinction to determine nickel. Then determine aluminium with 8-quinolinol by the extinction of a $CHCl_3$ solution. Finally determine manganese with $CHCl_3$ and sodium diethyldithiocarbamate at 578μ.'

Determination of Nickel in Tungsten Powder

K. L. ROHRER: 'Spectrophotometric Determination of Nickel in Tungsten Powder.'
Analytical Chemistry, 1955, vol. 27, July, pp. 1200–3.

Nickel, if present within the crystal structure of tungsten powder or as a dope, will alter the physical properties of the powder. The determination of nickel, in the small amounts in which it is likely to be found as an impurity, is a lengthy procedure and requires preliminary separation before a suitable colorimetric method can be utilized.

The author of this paper describes a rapid and accurate method by which the nickel is separated from all interfering elements by a chloroform extraction of the bivalent nickel complex of dimethylglyoxime, in the presence of the hydrogen peroxide used to dissolve the metal powder. The determination is made by spectrophotometric measurement of the diethyldithiocarbamate complex of nickel. Nickel, 1-50γ, can thus be determined in tungsten powder with an average deviation of less than 1γ.

Determination of Nickel in Malt Beverages

M. KENIGSBERG and I. STONE: 'Determination of Traces of Nickel in Malt Beverages.'
Analytical Chemistry, 1955, vol. 27, Aug., pp. 1339–40.

The report is one of a series covering investigations on the effects of trace metals in brewing processes and products.

The presence of nickel in worts and beers is stated to be assuming importance with the increasing use of nickel-containing materials in brewery equipment. It has been found that contamination of worts with a few p.p.m. of nickel may lead to toxic effects on the yeast and slowing of fermentation. Traces of nickel in the finished beer tend to form gas-evolving nuclei, resulting in a condition of 'wildness' or 'gushing beer'. (The results of the investigations leading to these conclusions are to be published shortly, in the *Proceedings of the American Society of Brewing Chemists.*)

The present paper gives details of technique developed in the Wallerstein Laboratories, New York

for determination of nickel in the amounts likely to be found in malt beverages of various types. The procedure described involves ashing of the sample, taking up the ash with dilute hydrochloric acid, and shaking out with dithizone in carbon tetrachloride. The soluble, coloured, nickel complex is formed in the aqueous layer by treatment with dimethylglyoxime, bromine water and ammonia, and the intensity of colouration is proportional to the amount of nickel present. In order to estimate the precision and reliability of the method, a series of tests was made in which recovery of added nickel was measured, and in this connexion the possible interference of copper and iron was investigated (copper 2 p.p.m., iron 5 p.p.m.). The data obtained in these experiments show that excellent recovery of nickel may be expected by this method, and that there is no interference from copper and iron at levels much beyond those which would normally be encountered.

ELECTRODEPOSITION AND OTHER COATING METHODS

Corrosion Testing of Electroplated Coatings

W. L. PINNER: 'Progress Report on Accelerated Corrosion Tests for the Performance of Plated Coatings.' *Plating*, 1955, vol. 42, Aug., pp. 1039–43. Report issued under the aegis of American Electroplaters' Research Project No. 15.

This is complementary to the First Progress Report, issued in 1953 (*Proc. Amer. Electroplaters' Soc.*, 1953, pp. 148–52: *Nickel Bulletin*, 1954, vol. 27, No. 2, pp. 24–5).

In the first report the investigators outlined the methods which were to be studied with a view to their suitability for adoption as accelerated tests for evaluation of the corrosion-resistance of metal coatings. The second report provides a critical assessment of 20 methods which have been examined. The broad nature of these tests, and the extent to which they are considered promising or of little potential value, is indicated below:—

1. Salt-spray or humidity test at low temperatures, in presence of corrosive gases such as SO_2. Discontinued, as unpromising.
2. Continuous immersion in solutions representative of conditions encountered in service. Further work deferred; immersion tests considered unpromising, on production parts, on account of galvanic effects on unprotected areas.
3. Exposure to corrosive solutions, followed by exposure in a humidity cabinet, with or without presence of corrosive gases. Method considered promising; work being actively pursued. See also tests 18 and 20.
4. Chilling, followed by spraying with dust-containing solution simulating early rain, and return to chilling chamber. Method holds little promise. A modification of this method, comprising chilling, rain spraying, and humidity exposure at high temperatures, gave more satisfactory results, but work is deferred pending results of tests which appear more promising.
5. Modification of the salt-spray test, e.g., intermittent operation, substitution of O_2 or CO_2 for air, or adjustment of pH by acids other than acetic. Discontinued in favour of other tests.
6. Addition, to the test environment, of solid particles such as dust, ash, soot. Found to be highly corrosive, and difficult to control. Likely to create weakness in coatings, rather than reveal existing faults. Discontinued.
7. Use of gelatin film to hold corrosive media in contact with coating while it is subjected to salt spray or other corrodent. Unpromising results; tests discontinued.
8. Exposure to moist atmosphere carrying sooty smoke, followed by exposure to unsooty moist atmosphere. Extremely corrosive, difficult to control. Discontinued.
9. Determination of effects of temperature on galvanic potential relationships. Discontinued, since the data do not give an actual measure of quality.
10. Study of influence of wetting agents on penetration of corrosive media into pores or cracks. Negative results obtained: test discontinued.
11. Determination of amount of current required to prevent corrosion in given test media. Results give no measure of quality; tests discontinued.

12A. Study of elimination of positional effects in spray boxes, by moving specimens and/or nozzles. Deferred for possible incorporation in general examination of spray tests.

12B. Investigation of effects of oxidizing agents other than air, in promoting breakdown of coatings in given media. Deferred for incorporation in other more general tests.

12C. Investigation of waterline effects in connexion with immersion tests. Deferred until immersion tests are considered worth further study.

13. Hot-water immersion tests, continuous or cyclic. Deferred in favour of more promising tests.
14. Acetic-acid modification of salt-spray test. Being actively studied, as giving promising results and reproducibility of service behaviour. Panels illustrating the behaviour of coatings so tested, in comparison with those subjected to outdoor exposure, are shown in the report.
15. Cyclic tests involving various periods of salt spray, infra-red exposure, chemical immersion, etc. Deferred in favour of simpler tests requiring less elaborate equipment.
16. Electrolytic corrosion test, consisting in making the part under test anodic in a dilute salt solution and with an impressed voltage lower than that of the solution potential of the metal involved. (For full details of test see *Proc. Amer. Electroplaters' Soc.*, 1954, pp. 176–84: *Nickel Bulletin*, 1954, vol. 27, No. 12, p. 226.) Test found to be active, but applicable in research work only.
17. British sulphur-dioxide test, comprising suspension of test sample in a closed container, over warm water to which SO_2 solution is periodically added. Test found to be active. Already in use in England: not yet fully studied in U.S.A.

18. Variation of test 3. Comprises road-slush splash test, followed by exposure in humid atmosphere. The chamber devised for application of the splash test (simulating conditions on city roads in winter) is described. Basic liquid is usually salt solution, to which clay soils, sand, and even pebbles may be added. Highly corrosive effects obtained: practical utility of test under close study.
19. Test developed in Germany, comprising exposure, in closed container, in atmosphere of SO_2, CO_2, and water vapour. Already in use in Europe; under active investigation in U.S.A.
20. Further variation of test 3, comprising application, by rubbing, of a synthetic soil to the plated surface, followed by exposure to a humid atmosphere. The synthetic corrodent, in paste form, is known as Corrodkote: a composition quoted as typical comprises kaolin 30 g., sodium chloride 15 g., ammonium chloride 15 g., with 50 ml. of water containing 2 g./L. of potassium fluoride. Various modifications have been used, to simulate individual sets of conditions to which coatings may be subject: some details of such variants are given, and the salient features of the humidity chamber employed in this test are outlined. The results are promising, and the method is under active study.

Cleaning of Metals Prior to Electroplating

H. B. LINFORD and P. E. GRUBB: 'Cleanability and Oil Spreading Rates.' *Plating*, 1955, vol. 42, July, pp. 895–902.

This report, presented at the 1955 Convention of the American Electroplaters' Society, is one of a series issued under the aegis of A.E.S. Research Project No. 12.

The object of this section of the work was to develop a correlation between the spreading rate of various oils, fats and greases, on a particular metal, with the degree of difficulty involved in cleaning that form of soil from another metal. The work was done as an introduction to spreading-rate studies on corrosion-resisting steel. Previous work had shown good correlation between cleanability and oil-spreading rate for oils which were in the liquid state at room temperature: in the present work the oils, fats and greases were of types solid at that temperature.

Production of Nickel by Electrolysis of a Chloride Solution

W. A. WESLEY: 'Preparation of Pure Nickel by Electrolysis of a Chloride Solution.' *Jnl. Electrochemical Soc.*, 1955, vol. 102, Sept., p. 228C.

Abstract of paper presented at the Annual Meeting of The Electrochemical Society, October, 1955.

'Pure nickel was prepared in the form of electrodeposits 3–5 mm. thick, from carefully purified nickel chloride-boric acid solution, using iridium-platinum alloy anodes. The overall reaction is

$$NiCl_2 = Ni_{(metal)} + Cl_{2\,(gas)}$$

As electrolysis proceeded, purified chloride solution was added automatically, to maintain the metal content of the electrolyte. The gas content of the metal is not known, but the total of spectrographically detectable impurities is only 34 p.p.m. The metal is malleable. Some data on mechanical and physical properties are included.'

Electrodeposition of Nickel from Fluoborate Solutions

C. B. F. YOUNG and W. STROBACH: 'Electrodeposition of Nickel from Fluoborate Solutions.' *Metal Finishing*, 1955, vol. 53, July, pp. 44–50; Aug., pp. 53–8; Sept., pp. 79–85.

Following a brief historical review of the development of fluoborate baths for electrodeposition of metals, the authors make a detailed report of an experimental investigation on deposition of nickel from such solutions. Attention was directed to the effect of bath composition and concentration (50–120 g./L. of metallic nickel), temperature, current density, pH, agitation, and the use of addition agents. The nature of the deposits produced, as a function of the respective variables, is reported, and some data are provided on hardness, ductility, uniformity and buffability of the coatings. Comparison was made with deposits obtained, under conditions as closely similar as possible, from (1) a Watts-type nickel bath and (2) a commercial bright-nickel solution of organic type. Both carbon steel and brass were used as cathodes. Throwing and covering powers of the respective solutions, economic aspects involved, and the suitability of the deposits as bases for chromium plating were also taken into consideration. Methods for analytical control of fluoborate baths were also studied.

A report is also made of some preliminary tests on nickel-cobalt fluoborate solutions, but this work is to be amplified in later investigations.

From the very detailed experimental data presented, the authors draw a number of practical conclusions and make recommendations for operation of fluoborate baths. The main points are summarized below:—

The presence of chloride in the fluoborate bath is beneficial, increasing both anode and cathode efficiencies and preventing gassing at the higher current densities. Nickel-fluoborate baths should contain at least 15 g./L. of nickel chloride.

Excess of free boric acid is advantageous, in stabilizing the solution and preventing formation of fluorides. Inclusion of at least 30 g./L. of free boric acid is recommended.

For general purposes a nickel concentration of about 80 g./L. is recommended, with an operating temperature of about 60°C. and pH about 3·0 colorimetric. Such conditions will permit the use of cathode current densities up to about 200 amp./sq. ft., with anode and cathode efficiencies approaching 100 per cent.

Pitting is a constant source of trouble in deposits made from fluoborate baths. Use of a wetting agent, to control pitting, is desirable; sodium *n*-octyl sulphate is specifically recommended. Surface tension of

the solution should be maintained at about 35 dynes/cm. when measured at 25°C.

The fluoborate bath is stable and, when operated under the conditions recommended, is readily kept in balance. pH tends to increase slightly during operation, but can be adjusted by addition of fluoboric acid.

Limiting current densities were established as a function of the metal content of the solution, and its temperature. With fluoborate baths it was possible to use current densities 2–4 times as great as those normally employed with nickel-sulphate solutions of comparable metal content.

Cathode efficiency increased with increasing current density below the limiting value, and with increasing pH in the range 1–2 colorimetric.

Limiting current density is increased by cathode agitation; some typical values are given.

Throwing power of nickel-fluoborate solutions compared favourably with that of sulphate baths; in all cases it decreased with increasing current density.

In impact tests deposits made from fluoborate solutions at a pH 3·0 colorimetric and above compared favourably, with respect to ductility and adhesion, with those produced from a Watts type of bath. As gauged by this test, nickel deposited from bright sulphate solutions proved relatively brittle.

Deposits from the fluoborate baths made under correct conditions of operation proved to be easily buffable, and readily accepted chromium plating.

Bright or semi-bright deposits were obtained from fluoborate solutions containing saccharin or para toluene sulphonamide, or a combination of the two. The brightest coatings were those made on smooth buffed brass, from a bath containing para toluene sulphonamide as the only brightener. Anode and cathode efficiencies and throwing power were not impaired by the use of this brightener, and the deposits remained ductile.

On deposits 0·005-in. thick made from fluoborate solutions the Knoop hardness numbers ranged from about 260 to 580, using a 200-gram load. The hardness rose as the current density and pH were raised, or as the temperature was lowered. The Knoop hardness of bright or semi-bright deposits from a fluoborate solution containing 80 g./L. of metallic nickel and addition agents was similar to that of deposits made from a bright sulphate solution of comparable metal content.

Although the cost of the fluoborate bath is fairly high, other considerations indicate that the use of such solutions is an economic proposition.

Electrodeposition of Molybdenum-Nickel Alloys

D. W. ERNST, R. F. AMLIE and M. L. HOLT: 'Electrodeposition of Molybdenum Alloys from Aqueous Solutions.' *Jnl. Electrochemical Soc.*, 1955, vol. 102, Aug., pp. 461–9.

Nickel-molybdenum, cobalt-molybdenum and iron-molybdenum alloys were electrodeposited from aqueous solutions containing sodium molybdate, the sulphate of the co-deposited metal, sodium citrate and ammonium hydroxide. Typical baths contained 0·3 M./L. of the respective alloy metals, 0·3 M./L. of sodium citrate, varying amounts of sodium molybdate, and ammonium hydroxide to secure a pH of about 10·5.

The maximum amount of molybdenum found in the deposit was dependent on the nature of the alloying metal involved: nickel alloys deposited from baths of the typical compositions referred to above contained up to 20 per cent. of molybdenum, cobalt alloys up to 40 per cent. molybdenum, and iron alloys about 50 per cent. molybdenum. The cathode current efficiencies in the three cases ranged, respectively, from 75 to 85, 50 to 60, and 10 to 20, per cent. The report includes observations made on the effects of pH, concentration, temperature and cathode current density, on cathode current efficiency and composition of the alloys deposited.

The electrodeposited alloys were in most cases metallic, though in the iron-molybdenum alloys they were covered in the centre with a dark black material believed to be a molybdenum oxide. They were either bright or light grey in appearance, and a large number of cracks were found in the bright deposits. When well-cleaned flat cathodes were used, good adhesion was obtained, but on tubing or rod adhesion was poor, and the deposits could in most cases be brushed off in flake or powder form.

The results presented lead the authors to conclude that development of an aqueous plating bath for electrodeposition of molybdenum alloys would be a practical proposition.

Activation of Non-Metallic Surfaces for Electroless Plating

F. PEARLSTEIN: 'Electroless Nickel Deposition: Activation of Non-Metallic Surfaces.' *Metal Finishing*, 1955, vol. 53, Aug., pp. 59–61.

Deposition of nickel by the electroless process, as developed by Brenner and Riddell, takes place only on certain metallic surfaces: the catalytic metals known to initiate deposition are iron, nickel, cobalt, aluminium, palladium, and, in some conditions, silver and gold. Since nickel itself is a good catalyst, once the initial nickel deposit is obtained on a surface deposition will continue as long as the solution is workable.

In order to deposit nickel by the electroless process on non-catalytic surfaces one of the following pretreatments must be employed:—

(1) Dipping in a solution of palladium salts, to produce a coating of palladium on the surface. This method is particularly useful for plating of copper and brass.

(2) Non-catalyst metals such as copper, brass and silver can also be plated by contacting the surface with a more electronegative metal, e.g., aluminium, while immersed in the electroless nickel solution. It is presumed that galvanic action initiates deposition.

(3) Initiation of deposition by application of an external source of current, to produce a thin coating of nickel, which will then act catalytically to ensure continuation of deposition by immersion.

The present article deals with experiments made to produce a thin film of palladium on non-conducting surfaces (mostly sheet polystyrene). It was found that simple immersion of this and other non-metallic materials for two minutes in 0·1 g./L. $PdCl_2$ at 125°F. (52°C.), pH 3·8–4·8, produced an active surface susceptible to coating with nickel by the electroless method. In an endeavour to find an improved method, consideration was given to the use of preliminary treatment in stannous chloride, which has the well-known property of being readily absorbed on many materials and is also a strongly reducing agent.

These trials showed that, in coating polystyrene, if a pre-dip in $SnCl_2$ solution is used the $PdCl_2$ bath can be used at room temperature and within a wider pH range (0·9–4·2). There are indications that several other non-metallic materials can be treated on the same principle, possibly with slight modifications of solutions and procedure according to the precise nature of the base.

NOTE.—A process claimed to be successful for electroless deposition of chromium is described in *Metal Finishing*, 1955, vol. 53, July, pp. 62–3.

NON-FERROUS ALLOYS

Reference Data on Metals and Alloys

See abstract on p. 174.

Nickel-Alloy and Other Thermocouples: Reference Data

See abstract on p. 174.

Specifications for Copper-Nickel Alloys and Stainless Steels Used in Pressure Vessels

See abstract on p. 192.

Heat Capacities of Gold-Nickel Alloy

'Calorimetric Investigations of a Gold-Nickel Alloy.' W. DESORBO: 'I. Low-Temperature Heat Capacity of Gold-Nickel Alloy.' *Acta Metallurgica*, 1955, vol. 3, May, pp. 227–31.

R. A. ORIANI: 'II. The Heat Capacity at High Temperatures and the Entropy of Formation', *ibid.*, pp. 232–5.

I. The heat capacity of gold-nickel alloy containing 52 at. per cent. of gold was measured in the region 13° to 300°K. (−260° to + 27°C.). It is shown that below 300°K. (27°C.) the experimental data do not conform to the Kopp-Neumann rule for additivity of heat capacities. The excess entropy, arising chiefly from the change in the vibrational spectrum upon alloy formation, is $0{\cdot}62\pm0{\cdot}09$ cal./g.-atom/° at 300°K. (27°C.). The values of the thermodynamic functions, entropy, enthalpy and free energy were evaluated and tabulated at regular intervals of temperature up to 300°K. (27°C.), for the 52 at. per cent. gold alloy. The entropy of the alloy at 298·16°K. (25°C.) is $9{\cdot}83\pm0{\cdot}07$ cal./g.-atom/°, of which 0·10 was obtained by extrapolating below 13°K. (260°C.).

II. The author reviews the literature on the measurement of heat capacity of alloys. Measurements were made of the cumulative enthalpy difference between the solid solution $Au_{0{\cdot}517}Ni_{0{\cdot}483}$ and the corresponding weights of the pure metals; differentiation of the temperature curve yields the deviation of the heat capacity of the alloy from the Kopp-Neumann rule. The positive value of this deviation and the large magnitude of the vibrational entropy of solution are related to the loosening of the lattice that accompanies formation of the solid solution.

Thermal Conductivities of Metallic Materials at Sub-Zero Temperatures

See abstract on p. 175.

Titanium-Nickel Alloys

D. H. POLONIS and J. G. PARR: 'Isothermal Decomposition Kinetics of Transformed-Beta Phase in a Titanium-Nickel Alloy.' *Acta Metallurgica*, 1955, vol. 3, July, pp. 307–11.

The tempering kinetics of some important ferrous alloys have been explored in detail by several investigators by applying theories of diffusion and precipitation developed by JOHNSON and MEHL (*Trans. Amer. Inst. Min. Met. Eng.*, 1939, vol. 135, p. 416) and ZENER (*Jnl. Applied Physics*, 1949, vol. 20, p. 950). The work has included decomposition studies of retained and martensitically transformed austenite, as well as of graphitization in cast irons. Most of the research has been concerned with interstitial alloying elements in materials of complex commercial composition.

In the present paper the authors make an analysis of the tempering kinetics of transformed-β solid solution (hexagonal close-packed) in a 7·2 wt. per cent. nickel titanium-nickel alloy. The work comprised study of growth of plates of Ti_2Ni during isothermal heat-treatments at temperatures between 450° and 550°C. It was found that at temperatures above 525°C. there was a tendency for the precipitation of thicker units and eventual formation of spheroids. The reaction, to 85 per cent. completion, is satisfactorily described by the rate equation based on work by JOHNSON and MEHL, COHEN, and ZENER. On the basis of the model proposed for the reaction and the activation energy of the process, it appears that self-diffusion of titanium is the major rate-controlling factor.

Welding of Copper and Copper-base Alloys

I. T. HOOK: 'The Welding of Copper and Its Alloys.' *Welding Jnl.*, 1955, vol. 34, July, pp. 321S–37S.

Survey of information contained in the literature in the English language published from 1936 to 1954. The review is complementary to that published *ibid.*, 1937, vol. 16, Mar. *Res. Suppl.*, pp. 7–46; it deals with the metallurgy of the copper alloys as

affecting the quality of welds, and with the effect of various joining methods, indicating the procedures which have proved most satisfactory in service. The materials covered include pure copper, the common brass and bronze alloys, (including nickel-aluminium bronzes), cupro-nickels and nickel silvers. The associated *bibliography* numbers 144 items.

Descaling of High-Temperature Alloys

See abstract on p. 195.

NICKEL-IRON ALLOYS

Sulphur Activity in Liquid Iron-Nickel Alloys

J. A. CORDIER and J. CHIPMAN: 'Activity of Sulphur in Liquid Fe-Ni Alloys.' *Jnl. of Metals*, 1955, vol. 7, Aug.; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, vol. 203, pp. 905–7; T.P. 4049C.

Activity of sulphur in liquid iron was determined in 1950 by SHERMAN *et al.* (*ibid.*, vol. 188, p. 334; *Jnl. of Metals*, Feb., 1950), and in that paper a summary was made of data resulting from the work of other investigators on the effects of a number of alloying elements on the activity of sulphur in iron. The results were obtained by a study of the equilibrium in the reaction of sulphur in the liquid metal with hydrogen gas, to form H_2S.

The present paper reports experiments made by the same method, from which it is concluded that the chemical behaviour of sulphur in liquid steel is independent of the nickel content, and that sulphur has the same activity and free energy in liquid nickel and iron-nickel alloys as in liquid iron.

The influence of other elements upon the reactions of sulphur in iron and nickel is also discussed, and in this connexion the following conclusions are considered to be justified:—

'A number of metals, which are typified by manganese, chromium, and cobalt, show no specific reactions in the liquid state with either iron or nickel and are presumed to form nearly ideal solutions in either metal. The effect which such an element exerts upon sulphur is likely to be very much the same in liquid nickel as in liquid iron. Carbon, oxygen, aluminium and silicon, however, behave rather differently in iron and in nickel, and their specific effects upon sulphur may be expected to be different in the two solvent metals. Magnesium and calcium are much more soluble in liquid nickel than in iron and they are therefore used much more effectively as desulphurizers in nickel than in iron.'

Self Diffusion of Iron in Nickel-Iron Alloys containing Carbon

P. L. GRUZIN and E. V. KUZNETSOV: 'Influence of Carbon on the Self-Diffusion of Iron in the Iron-Nickel System.' *Metal Progress*, 1955, vol. 68, Aug. 1, pp. 176, 178.
Abstract of report in *Doklady Akad. Nauk.*, U.S.S.R., 1953, vol. 93, pp. 809–12.

In previous studies of self-diffusion of iron in austenite the senior author had concluded that the binding of the iron atoms decreased with increase in carbon content. The work abstracted here was designed to investigate this aspect more closely, in a binary iron-base alloy. The range of composition used was nickel 19·65–25·0, carbon 0·02–0·95, silicon trace – 0·1, manganese trace – 0·19, per cent., balance iron. Details of experimental technique are given.

The results indicate that 'carbon decreases the binding of iron to the iron-nickel solid solution and that the higher the nickel content of the alloy the smaller is this effect.'

Reference Data on Metals and Alloys

See abstract on p. 174.

Magnetization Reversal in Thin Films of Nickel-Iron Alloy

R. L. CONGER: 'Magnetization Reversal in Thin Films.' *Physical Rev.*, 1955, vol. 98, June 15, pp. 1752–4.

It has been shown that magnetization reversal in single crystals having dimensions of the order of a centimetre takes place by the motion of 180° domain walls, and that the wall velocity is controlled by eddy-current effects. It is believed that in most polycrystalline materials magnetization reversal takes place by that process. If, however, a ferromagnetic film is too thin to support domain walls, the reversal would be expected to take place by a different mechanism.

This paper reports experiments on evaporated films (2×10^{-5} cm. thick) of an alloy containing nickel 80, iron 20, per cent. The results demonstrate that magnetic reversal takes place by domain rotation rather than by the motion of 180° domain walls.

Texture/Thickness Relationships in Magnetic Nickel-Iron Alloys

S. SPACHNER and W. ROSTOKER: 'On the Loss of Texture in Tapes of a 50%Ni-50%Fe Alloy.' *Jnl. of Metals*, 1955, vol. 7, Aug.; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, vol. 203, pp. 921–2; T.N. 271E.

A 50–50 nickel-iron alloy is currently used for production of high-permeability magnetic tape, and in the annealed condition this alloy produces an almost square hysteresis loop, which has been attributed to the fact that by correct rolling and annealing procedure a sharp cube (100) [001], texture can be developed.

This note records experiments made as part of a general study of the relation between heat-treatment, magnetic properties and structure in tape core materials. Annealed tapes of the 50–50 alloy were examined in 2, 1, $\frac{1}{2}$, $\frac{1}{4}$ and $\frac{1}{8}$ mil. gauges. Pole figures constructed for the 2- and 1-mil tapes showed the expected sharp cube texture, but in the thinner tapes there was no evidence of any preferred orientation.

The work recorded shows that there is a sharp change in the annealing characteristics of tapes of alloy of this composition between thicknesses of

1 and ½ mil. Above 1 mil a sharp cube structure can be produced: below that thickness no preferred texture can be developed. The occurrence of this effect is apparently not dependent on heat-treatment practice, and is not due to a size effect on recrystallization. There is no conclusive evidence that it is related to the structural conditions obtaining in the surface of the tape as a result of the complex strain relationships developed in the rolling process. Diffraction studies on tapes of Permalloy and nickel (heavily cold-rolled and annealed), in thicknesses below 1 mil, also showed an absence of preferred orientation, indicating that the effect observed in the 50–50 nickel-iron alloy may be more general than had been supposed.

Grain-Boundary Effects in High-Permeability Nickel-Iron Alloys

R. E. S. WALTERS: 'Grain Boundaries in Alloys of High Magnetic Permeability.'
Acta Metallurgica, 1955, vol. 3, May, pp. 293–4.

The development of high magnetic permeability in nickel-iron-base alloys containing molybdenum and copper (of the Mumetal or Permalloy C types) is achieved by annealing in pure hydrogen at temperatures above 1000°C. The functions of this treatment are believed to be (*a*) mechanical softening through recrystallization and grain growth, and (*b*) removal of impurities such as carbon, nitrogen and oxygen.

The belief that grain boundaries are regions of relatively low permeability is supported by the observation that highest permeabilities are developed in specimens which have been annealed at the highest temperatures, i.e., those having the largest crystals. More precise data relating magnetic behaviour with grain size are, however, required. This note reports experiments with specimens of an alloy containing nickel 77, iron 14, copper 5, molybdenum 4, per cent., prepared from powders, rolled under controlled conditions, and annealed at various temperatures to produce varying grain size. Initial permeabilities were determined as a function of mean grain diameter. The predicted linear relation was established, and the observations made support the assumption that grain-boundary areas are of relatively low permeability.

Permalloy Tape Cores in Digital Computers

N. MENYUK: 'Magnetic Materials for Digital Computer Components. II. Magnetic Characteristics of Ultra-Thin Molybdenum-Permalloy Cores.'
Jnl. Applied Physics, 1955, vol. 26, June, pp. 692–7.

Recent studies in the Massachusetts Institute of Technology have led to the conclusion that ultra-thin 4–79 molybdenum-Permalloy tape cores are superior to ferrites for use in switching circuits and stepping registers. Among the factors favouring the metallic tapes are their superior thermal properties, higher flux density, and more rapid flux-reversal characteristics. (For details of the work on which these conclusions are based, see *Trans. Amer. Inst. Electrical Engineers, Commn. and Electrical Section*, 1955, vol. 16, p. 631.)

In the present paper the author reports measurements of flux-reversal time as a function of applied magnetic field, for ⅛, ¼, ½ and 1 mil 4–79 molybdenum-Permalloy tape cores. The measurements were made at seven temperatures, ranging from −196° to +270°C., and from them the switching coefficient is found. Determination of the switching coefficient as a function of tape thickness permits separation of the contributions to switching delay made, respectively, by spin relaxation and by eddy currents. These contributions have been studied individually in relation to temperature.

Oxidation Characteristics of Nickel-Iron Alloys

R. T. FOLEY: 'The Oxidation of Iron-Nickel Alloys.'
Jnl. Electrochemical Soc., 1955, vol. 102, Aug., pp. 440–5.

Iron and nickel form a continuous series of substitutional solid-solution alloys, both components of which are oxidizable. The kinetics of oxidation have already been investigated on alloys containing up to 30 per cent. of nickel, but no systematic study had previously been made of the behaviour of alloys containing about equal proportions of the two elements, in the 600°–900°C. region. In view of the extensive use of such alloys in making glass-to-metal seals, and in other applications in the electrical and electronic industries, some knowledge of their oxidation characteristics is to be desired.

The purpose of the work described in the present paper was a study of the kinetics of the alloy containing 42 per cent. of nickel, and elucidation of the mechanism of oxidation from the reaction rate and the nature of the surface layers. It was also considered advisable to assess the influence, on oxidation behaviour, of the surface condition of the alloys.

The specimens consisted of strips of alloy having an apparent surface area of 25·8 cm²., cut from a band which had been finally cold-finished to a thickness of 0·051 cm. The strips were annealed, and degassed in dry hydrogen for 2 hours at 1125°C., after which they were cooled in hydrogen to about 75°C. Provision was then made for four variations in surface finish:—

(1) '*As rolled*.' After annealing in hydrogen, as described above, specimens were swabbed with benzene and acetone, and dried before weighing. While these samples had a bright appearance, electron-diffraction examination showed the presence, in faint but sharp pattern, of Fe_2O_3.

(2) '*Chemically cleaned*.' The strip was pickled, at 90°C. for 2 minutes, in an aqueous sulphuric-nitric-hydrochloric acid solution; rinsed; dipped in a sulphuric-nitric acid mixture and again rinsed. The specimens were then allowed to stand for one hour in sodium-cyanide solution, to neutralize any residual acid, and were again rinsed. Final treatment was rinsing in acetone, in which medium the strips were kept until used for oxidation test.

(3) '*Sand-blasted*.' There was no evidence of sand remaining on the strips, but analyses made after

oxidation warranted the conclusion that the abrasive was embedded in the surface.

(4) '*Polished.*' Polished with three grades of paper, swabbed with benzene and acetone, and stored in acetone until used.

Oxidation tests were made in a resistance wire-wound furnace having a static air atmosphere: temperatures of test 600°, 700°, 800° and 900°C., for times varying up to 60 minutes.

Films stripped from the oxidized strips were chemically analyzed and examined by electron-diffraction and metallographic methods. The observations made show that, for the alloy examined, the temperature dependence of the parabolic oxidation-rate constant follows the Arrhenius equation, with the parameters dependent on the method of surface preparation. The oxide film, on cooling, shows a two-phase structure consisting of nickel ferrite ($NiFe_2O_3$) next to the metal, and Fe_2O_3. Differences in activation energies and oxidation rates are explained on the basis of diffusion through ferrite structures of varying nickel content.

CAST IRON

Ni-Resist: Properties and Applications

INTERNATIONAL NICKEL CO., INC.: 'Engineering Properties and Applications of Ni-Resist.'
4th (revised and enlarged) edn., published 1955; 64 pp.

This publication contains a comprehensive summary of information on the properties typical of the various grades of high-nickel cast iron in the Ni-Resist and Minovar groups, and a review of applications in numerous industries in which their characteristic combination of mechanical and corrosion-resisting properties is utilized. Since the material is essentially of a reference character, the information available in the publication is best indicated by a skeleton of the contents, which include particulars of the compositions and essential characteristics of this series of alloy cast irons, properties affecting design and manufacture (including reference to machinability, welding, and annealing procedure), mechanical and physical properties, wear- and galling-resistance, erosion- and corrosion-resisting qualities, heat-resistance, thermal-expansion properties, thermal conductivity, low-temperature properties, and electrical and magnetic properties.

The second section of the handbook is concerned with fields of application in which one or more of the Ni-Resist group of cast irons is in established use: these include chemical-process industries, food-handling equipment, internal-combustion engines, miscellaneous equipment used in handling corrosive liquids, items of marine equipment, plant used in various branches of the petroleum industry, parts of power plant, precision tools, equipment used in production of pulp and in paper-making, and general industrial equipment. The final section contains a comprehensive selection of corrosion data, based on results accumulated over a long period as a result of laboratory tests and service records. The behaviour of Ni-Resist cast irons is compared with unalloyed cast irons, in some 400 different conditions likely to be encountered in day-to-day commercial operation.

Annealing of S.G. Iron

G. N. J. GILBERT: 'A Comparison of the Microstructure and Mechanical Properties of Nodular Irons Ferritized by Two-Stage and Single-Stage Annealing Treatments.'
Brit. Cast Iron Research Assocn., Jnl. of Research and Development, 1955, vol. 6, Aug., pp. 11-15; *Research Rept. No.* 410.

In order to obtain high elongation in the tensile test, and good resistance to impact, pearlitic spheroidal-graphite cast irons are annealed to the ferritic condition. Theoretically, the treatment can be carried out according to either of two cycles:—

(a) holding at 900°C. for 4 hours; furnace-cooling to 690°C., and holding for 8 hours at that temperature, followed by slow cooling in the furnace;
(b) holding at 690°C. for about 8 hours and furnace-cooling.

The treatment at 900°C. is designed especially to eliminate free carbide, and if no carbide is present in the as-cast structure this part of the cycle is in some cases omitted and treatment at the sub-critical temperature (690°C.) only is applied, although it is admitted that the higher-temperature treatment permits homogenization of the matrix which is not effected at the lower temperature.

Conclusions reached as a result of work described in this paper (see below) are based on comparative mechanical tests and examination of microstructure of specimens from a single heat of S.G. iron (made by nickel-magnesium-cerium alloy) subjected to four different annealing treatments:—

(1) 14 hours at 950°C.; slowly cooled to 690°C.; held at 690°C. for 72 hours; furnace-cooled.
(2) 14 hours at 900°C.; slowly cooled to 690°C.; held at 690°C. for 72 hours; furnace-cooled.
(3) 14 hours at 850°C.; slowly cooled to 690°C.; held at 690°C. for 72 hours; furnace-cooled.
(4) 72 hours at 690°C.; furnace-cooled.

The data presented demonstrate that irons treated by cycle (b) above show a sub-boundary structure in the ferrite grains, which may be associated with brittleness. Irons treated by the double cycle (a), however, show satisfactory ferrite structures. Satisfactory ferritic structures are obtained whether the irons are normalized by air cooling from 900°C. to room temperature before treatment at 690°C., or simply furnace-cooled from 900°C. to the 690°C. annealing temperature. Tests on other S.G. irons confirmed the above conclusions.

Reference Data on Metals and Alloys

See abstract on p. 174.

CONSTRUCTIONAL STEELS

Sulphur Activity in Liquid Iron-Nickel Alloys

See abstract on p. 186.

Self Diffusion of Iron in Nickel-Iron Alloys containing Carbon

See abstract on p. 186.

Flake Formation in Steels

A. W. DANA, F. J. SHORTSLEEVE and A. R. TROIANO: 'Relation of Flake Formation in Steel to Hydrogen, Microstructure, and Stress.' *Jnl. of Metals*, 1955, vol. 7, Aug.; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, vol. 203, pp. 895–905; T.P. 3968 CE.

In spite of the large amount of research which has been devoted to the study of flaking (hairline cracking) in steel, no final conclusions have been reached as to its cause. Many theories have been advanced to explain the phenomenon: the main features of two of them, the hydrogen-pressure theory and the micro-segregation and transformation-stress theory, are outlined as an introduction to this report, and it is pointed out that both lines of argument appear to be compatible with industrial experience. The literature based on examination of laboratory and of commercially produced steels is, however, contradictory, and the present paper reports tests made in an endeavour to evaluate the respective effects of stress and of hydrogen.

The method adopted was to introduce into steels of medium-to-high hardenability, and remove from them, controlled amounts of hydrogen, followed by a transformation analysis of the steels. Experimental technique is fully described.

Preliminary experiments to evaluate the effect of flake formation in a steel of the S.A.E. 4340 type (carbon 0·4, nickel 1·75, chromium 0·85, molybdenum 0·25, per cent. approx.) were followed by confirmatory tests on low-alloy nickel, nickel-chromium, chromium-molybdenum and chromium steels, variously heat-treated, and with or without varying amounts of hydrogen. A highly detailed report, with much tabular and photomicrographic illustration, is given.

The very extensive data recorded are considered to demonstrate that flaking results from a combination of hydrogen and of stress: hydrogen sensitizes the steel to brittle behaviour. The stress pattern in a section therefore plays an important part, and it is, in turn, particularly sensitive to the state of transformation of austenite, i.e., the nature, distribution, and proportions of the microconstituents. Thus, the transformation characteristics of austenite are of primary significance.

The authors' observations on isothermally treated and/or continuously-cooled specimens of the low-alloy steels used indicate that hydrogen in the steel is a pre-requisite for flake formation, and that transformational stresses must also be present. Flaking does not occur if decomposition of austenite is completed above M_S prior to cooling to room temperature. Under appropriate conditions abnormally high hydrogen contents may be tolerated without occurrence of flaking.

The greatest tendency for flake formation is in mixed structures containing small amounts of martensite. There is no correlation between flake formation in such mixed structures and the average hydrogen contents.

Predominantly martensitic structures exhibit radial or quench cracks which are related to the geometry of the specimen. Cooling stresses are believed to play an important rôle, together with stresses arising from transformation, and average hydrogen content.

It would appear that flakes do not form in the absence of transformation stresses and that, at least where cooling stresses are low, formation of flakes probably cannot be correlated with average hydrogen content of the section if such contents are not excessively high.

Sub-Zero Tensile and Impact Properties of Plain and Low-Alloy Steels

H. F. HALL and R. W. NICHOLS: 'Yield Behaviour of Metals at Low Temperatures, with Particular Reference to Some Carbon and Low-Alloy Steels.' *Jnl. Iron and Steel Inst.*, 1955, vol. 180, Aug., pp. 329–36.

Although it is generally known that ferritic steels show a marked decrease in toughness with fall in temperature, most of the data available in the literature have been obtained by means of the notched-bar impact test, and but little correlation of these results with low-temperature tensile properties has been made. This paper reports comparison of notched-bar impact and slow-speed tensile tests, made on identical materials, using apparatus specially designed to facilitate study of the effect of temperature on the early stages of plastic deformation. Apparatus and testing technique are described and illustrated.

The materials tested comprised 0·15, 0·30, 0·55 and 0·70 per cent. carbon steels, three alloy steels containing, respectively, (*a*) carbon 0·6, manganese 1·2, per cent.; (*b*) carbon 0·35, nickel 3, chromium 0·7, per cent.; (*c*) carbon 0·39, nickel 5, per cent. The carbon steels were tested in the as-rolled, annealed, and quenched-and-tempered conditions; all the alloy steels were tested in the quenched-and-tempered conditions, and for the manganese and the 5 per cent. nickel steels annealed specimens were also used. Copper and an aluminium-base alloy were also included. The determinations were made at several temperatures within the range $+200°$ to $-197°$C.

The proof-stress values for the steels were found to increase markedly with lowering of temperature, especially in the lower ranges studied, and the smaller the room-temperature values the greater was the increase as the temperature of test was lowered. For most of the materials tested, fall in temperature also increased the difference between the upper and

the lower yield points, the yield extension, the elastic modulus, the ultimate stress and (except in the temperature range over which the tensile ductility decreased marked with fall in temperature) the breaking stress.

Correlation of tensile and impact properties showed that room-temperature reduction in area provides some index to the Charpy maximum energy of a material, but there was no direct relation between any of the tensile properties and the impact-transition temperatures. This lack of correlation is explained in terms of the flow and fracture stress concept of brittle fracture: dynamic factors calculated on this basis from the test results showed reasonable agreement with experimental data.

Thermal Conductivities of Metallic Materials at Sub-Zero Temperatures

See abstract on p. 175.

Influence of Normalizing and Tempering on High-Temperature Properties of Chromium-Nickel-Copper Steel

See abstract on p. 192.

Reference Data on Metals and Alloys

See abstract on p. 174.

Preheating for Prevention of Hard-Zone Cracking in Low-Alloy Steels

C. L. M. COTTRELL and B. J. BRADSTREET: 'Calculated Preheat Temperatures to Prevent Hard-Zone Cracking in Low-Alloy Steels.' *Brit. Welding Jnl.*, 1955, vol. 2, July, pp. 310–12.

This report is based on an investigation carried out by the British Welding Research Association: it was circulated earlier in 1955 to the members of the Association as a confidential report. The information is of a reference character, comprising tables (based on experimental data) which permit calculation of the preheat temperature required for metal-arc welding of various types of low-alloy steel. Selection is based on knowledge of the dimensions and form of the joint, the size of the weld, and the weldability index of the individual steels as gauged by the 'thermal severity number' derived from behaviour in the C.T.S. test assembly, for details of which see *Welding Jnl.*, 1953, vol. 32, pp. 257S–72S.

Among the low-alloy steels for which weldability indices are given, and to which the calculations in this paper may be applied, are types containing nickel in amounts varying up to 2·52 per cent.

Carbon and Low-Alloy Steel Arc-Welding Electrodes

AMER. WELDING SOC. and AMER. SOC. TESTING MATERIALS: 'Specifications for High-Tensile and Low-Alloy Steel Covered Arc-Welding Electrodes.' Published by *Amer. Welding Soc.*, *New York*. Price 40c.

The revised edition of this specification classifies the electrodes according to chemical composition, in addition to the classification by mechanical properties, as formerly used. The new procedure will permit the purchaser to select low-alloy steel electrodes which will not only provide the required mechanical properties in the weld, but will also ensure weld metal closely matching the parent materials in corrosion-resistance and other characteristics.

The specification also gives details of test methods and requirements, standard sizes and lengths, packaging and marking, together with general notes on interpretation and practical application of the requirements laid down.

Photometric Determination of Nickel in Steel

See abstract on p. 181.

HEAT- AND CORROSION-RESISTING MATERIALS

Machining, Finishing and Inspection of Nimonic Alloy Gas-Turbine Blades

'Rolls-Royce Dart. The Manufacture of Components for an Airscrew-Turbine Power Unit. Part I. Turbine Blades: Machining and Inspection Techniques.' *Aircraft Production*, 1955, vol. 17, Sept., pp. 340–50.

A brief note is given on the development of the Dart engine, which has been designed with specific reference to the need for the long life and consistent performance called for in civil aviation, e.g., in the Vickers Viscount aircraft. A series of articles will describe the manufacture of several of the main components of the engine, and, in view of the vital importance of the blades, they are selected for initial treatment. The turbines have a high-pressure and a low-pressure stage, and the sequence of manufacturing operations is similar for blades in both stages: it is described in this article as applying to the low-pressure blades.

The blades are made from Nimonic 80A (titanium-aluminium-containing nickel-chromium-base alloy), the full composition and properties of which are tabulated. A detailed description is given, with over twenty excellent illustrations, of equipment and methods involved in rough milling, grinding, broaching, milling of concave and convex contours, inspection, and polishing. *Inter alia*, particular attention is directed to the electrolytic polishing method used, which has resulted from co-operative work by Rolls-Royce and others. Final machining and finish-inspection are also described.

It is noted that, with a view to reducing the number of machining operations and man-hours required to produce the final form, a new method, based on close-forged blades, is under development by Rolls-Royce. Some of the main features of this process are briefly noted.

Deep Drawing of High-Nickel Alloys

'Deep Drawing of Nimonic 75 and Stainless Steel.' *Sheet Metal Industries*, 1955, vol. 32, Aug., pp. 572–5, 580.

This article contains an illustrated description of the 'Lancastrian' press which is in use at the works of Joseph Lucas (Gas Turbine Equipment), Ltd., for operations involved in production of annular combustion units for gas turbines. A step-by-step account of the working of the machine is given. A wide range of work is done on it, from single reductions on 45-in. diameter blanks, to six-stage reductions on 54-in. diameter blanks, in gauges from 21 to 14 s.w.g.

Nimonic Alloys in Propeller Gas Turbines

'Component Life in Propeller Turbines.' *Engineering*, 1955, vol. 180, Sept. 9, p. 328.

High-temperature components such as flame tubes and rotor blades of gas turbines were, in the early stages of development, regarded as necessarily short-lived, due to the severe conditions in which they operated. Striking extensions of life have, however, been achieved, and this short article gives some facts regarding life obtained, in regular service, with Nimonic 75 flame tubes and Nimonic 80A and 90 blades.

For example, when the Vickers Viscount airliner was introduced into the British European Airways service, in April 1953, the time between major overhauls of its Rolls-Royce Dart turbo-propeller engines was 400 hours, and the average life of the flame tubes was of about the same order. Replacements of flame tubes were therefore made as part of the regular overhaul. The overhaul period for the engines has gradually been lengthened, and the life of the flame tubes has been progressively raised to 1,050 hours. An immediate target of 1,250 hours for engine life, including flame tubes, has recently been set.

The Bristol Aeroplane Company, in developing the Proteus 705 and 755 turbo-propeller engines for the Bristol Britannia airliner, have found that at the end of stringent type tests Nimonic 75 flame tubes are in a condition warranting the expectation that when the Britannia goes into service with British Overseas Airways Corporation the overhaul life of the Proteus, including its flame tubes, will rise rapidly to about 1,000 hours.

Failure of turbine blades is very rare, and is usually traceable, when it does occur, to uneven temperature distribution in the combustion gases or to resonance caused by unsuitable blade design. In operation of the Viscount very little trouble has been experienced with the Nimonic 80A blades of the Dart engines: the longest period completed by any one set of high- and low-pressure blades is 1,800 hours (representing more than 500,000 miles flying), after which optical inspection indicated that the blades were fit for further use. Highly satisfactory results are also reported for the cast blades of Nimonic 80A and 90 which are being used in the Proteus engine.

Titanium-Carbide/Nickel-Alloy Cermets for High-Temperature Service

K. PFAFFINGER, H. BLUMENTHAL and F. W. GLASER: 'Titanium-Carbide-base Cermets for High-Temperature Service.' *Amer. Soc. Testing Materials, Preprint* 94b, June, 1955; 10 pp.

The paper deals with the production and properties of a group of materials comprising high-purity titanium carbide bonded with nickel-chromium or nickel-cobalt-chromium alloys. Such materials, as developed by Metallwerk Plansee, Reutte, are designated the WZ series. They are intended for service in the range 925°–980°C.

The compositions of the main groups in the series are shown in Table I below:—

Table I
Composition and Physical Properties of WZ Cermets

Designation	Composition				Density g. per c.c.	Vickers Hardness	Average Coefficient of Thermal Expansion per °C. 20°–1000°C.
	Titanium Carbide %	Nickel %	Cobalt %	Chromium %			$\times 10^{-6}$
WZ–1b ..	60	32	—	8	6·20	950	10·2
WZ–1c ..	50	40	—	10	6·50	790	—
WZ–1d ..	35	52	—	13	6·90	590	12·6
WZ–12a ..	75	15	5	5	6·0	1070	9·9
WZ–12b ..	60	24	8	8	6·25	960	9·2
WZ–12c ..	50	30	10	10	6·55	820	10·6
WZ–12d ..	35	39	13	13	6·95	600	11·9

It is stated that in both types it has been found desirable to incorporate small amounts of molybdenum, to eliminate free carbon and oxygen which, if present, would be deleterious.

Production of the composites is by normal powder-metallurgy techniques: sintering is carried out in a vacuum furnace equipped with molybdenum elements, and the vacuum obtained during treatment is of the order of 0·1 mm. of mercury.

Properties of the cermets are closely related to the nature and amount of the binder present; values stated to be typical for the respective grades are shown in Table II on page 193.

With regard to the potential applications of the composite materials, it is recorded that specimens representing actual engine components have recently been made and that special test methods are being devised in order to examine exhaustively the behaviour of the carbide/metal materials under severe conditions at high temperatures.

The properties of the WZ series of high-temperature cermets are described also in a paper by K. PFAFFINGER entitled 'Titanium-Carbide-base Hard Metals for High-Temperature Service', contained in *Planseeberichte Pulvermetallurgie*, 1955, vol. 3, No. 1, pp. 17-33.

High-Temperature Properties of Brazed Joints in Nickel-containing Alloys

C. A. GYORGAK and A. C. FRANCISCO: 'Preliminary Investigation of Properties of High-Temperature Brazed Joints Processed in Vacuum or in Molten Salt.' *Nat. Advisory Committee for Aeronautics, Tech. Note* 3450, May, 1955; 29 pp.

The tests reported covered determinations of the shear strength of brazed joints made in various materials, using Nicrobraz (nickel-chromium-base alloy containing 4·9 per cent. boron, and 4 per cent. silicon), with or without modification by nickel additions.

The materials brazed were typical nickel-, cobalt- and iron-base alloys used in high-temperature applications, and the variables studied were the effect of method of brazing (molten salt bath or vacuum furnace), the effect of nickel additions to the brazing alloy, brazing temperature, (1950°, 2050° and 2150°F.: 1065°, 1120° and 1175°C.), time of holding at brazing temperature (2, 6 or 10 minutes), and the effect of a protective coating on the brazing area in specimens brazed in salt-bath conditions.

The results of the tests indicate that both methods of brazing are capable of producing satisfactory joints, having shear strength of the order of 63,000 p.s.i. (28 tons per sq. in.) for vacuum-made joints and 48,000 p.s.i. (21·5 tons per sq. in.) for salt-bath brazing. The strengths obtained are higher than those which have been reported for joints processed in dry hydrogen. In brazing in molten salt baths the shear strength of the joint increases as the temperature of brazing is raised, and the use of a protective coating increased the strength of the joints and improved reproducibility of results. In vacuum processing temperature of brazing and time at temperature were found to be the important variables; the best results were obtained by 15 minutes at 2075°F. (1135°C.). The addition of 10, 30 or 50, per cent. of nickel to the brazing alloy had no significant effect on the strength of the vacuum-processed joints, but was slightly beneficial when the salt-bath process was used.

Specifications for Copper-Nickel Alloys and Stainless Steels Used in Pressure Vessels

'Proposed Revisions and Addenda to A.S.M.E. Boiler and Pressure-Vessel Code.'
Mechanical Engineering, 1955, vol. 77, June, pp. 546-7.

As need arises in industry, the Boiler and Pressure-Vessel Committee of the American Society of Mechanical Engineers considers suggestions for revisions in the Code. After scrutiny, and possible amendment by the Committee, the proposed modifications are published, for comment, in *Mechanical Engineering*, which is one of the organs of the Society. After final approval such addenda or modifications are embodied in the Code.

In the schedule referred to above proposals are made for:—

(*a*) stress values, at various temperatures up to 700°F. (370°C.), for seamless pipes and tubes in three grades of copper-nickel alloy (70-30, 80-20 and 90-10);

(*b*) stress values, at various temperatures up to 1500°F. (815°C.), for seamless pipes and tubes made from five grades of austenitic chromium-nickel steel.

Both items are addenda to be incorporated in existing specifications under the jurisdiction of the Boiler Committee.

Oxidation of Nickel: Initial Stages

See abstract on p. 179.

Oxidation Characteristics of Nickel-Iron Alloys

See abstract on p. 187.

Ni-Resist: High-Temperature Properties and Applications

See abstract on p. 188.

Thermal Conductivities of Metallic Materials at Elevated Temperatures

See abstract on p. 175.

Influence of Normalizing and Tempering on High-Temperature Properties of Chromium-Nickel-Copper Steel

M. H. JONES, D. P. NEWMAN, G. SACHS and W. F. BROWN: 'Effects of Variation in Normalizing and Tempering Procedure on Stress-Rupture Strength, Creep Embrittlement and Notch Sensitivity for a Cr-Mo-V and a 17Cr-4Ni-Cu Steel.'
Trans. Amer. Soc. Metals, 1955, vol. 47, pp. 926-54; disc., pp. 954-6.

The tests described in this paper were made on steels of the compositions shown on page 194.

Table II

Strength Properties of WZ Cermets

Designation	Binder %	Flexural Strength				Tensile Strength				Charpy Impact Strength ft.-lb.		Modulus of Elasticity, at 20°C.	Stress Rupture 100-hour Life			
		20°C.		900°C.		20°C.		900°C.					800°C.		1000°C.	
		p.s.i.	t.s.i.	p.s.i.	t.s.i.	p.s.i.	t.s.i.	p.s.i.	t.s.i.	20°C.	900°C.	p.s.i.	p.s.i.	t.s.i.	p.s.i.	t.s.i.
WZ-1b	40	185,000-199,000	82·5-89·0	—	—	99,000-114,000	44·0-51·0	64,000	28·5	3·16	4·16	54,500,000	—	—	—	—
WZ-1c	50	226,000-242,000	101·0-108·0	—	—	127,000-142,000	56·5-63·5	72,000	32·0	4·16	6·47	—	—	—	—	—
WZ-1d	65	242,000-255,000	108·0-114·0	—	—	135,000-150,000	60·5-67·0	60,000	27·0	6·56	—	—	—	—	—	—
WZ-12a	25	170,000-185,000	76·0-82·5	—	—	85,000- 99,000	38·0-44·0	—	—	2·73	3·09	59,500,000	—	—	—	—
WZ-12b	40	191,000-213,000	85·5- 95·0	—	—	114,000-127,000	51·0-57·0	71,000	31·5	3·09	—	56,000,000	—	—	15,500	7·0
WZ-12c	50	226,000-255,000	101·0-114·0	73,000	32·5	127,000-142,000	57·0-63·5	64,000	28·5	4·53	5·83	50,700,000	45,500	20·5	12,500	6·5
WZ-12d	65	248,000-268,000	111·0-120·0	68,000	30·5	142,000-156,000	63·5-69·5	54,000	24·0	7·00	8·71	46,000,000	37,000	16·5	9,500	4·5

Steel	C %	Si %	Mn %	Cr %	Ni %	Cu %	Mo %	Fe %	Other Elements %
17–22 A(S) ..	0·33	0·68	0·57	1·23	0·23	0·16	0·53	Bal.	V 0·24
17–4 PH ..	0·05	0·33	0·64	17·86	4·43	3·91	—	Bal.	Nb 0·36

Although it is generally recognized that creep strength of low-alloy steel is sensitive to heat-treatment, there are few data on the influence of thermal treatment on high-temperature stress-rupture, and particularly on creep embrittlement. Relevant literature is reviewed.

Earlier work by the present group of authors had given some indications of the trend of creep-resistance, rupture-strength and ductility likely to be associated with variations in heat-treatment, and in the light of those findings the low-alloy chromium-molybdenum-vanadium and the ferritic precipitation-hardening stainless steel were selected for detailed study of the following aspects:—

(*a*) influence of normalizing temperature on conventional (smooth) rupture properties and on notch rupture,

(*b*) influence of tempering temperature and time on smooth and notch-rupture properties,

(*c*) possible relationships between time-temperature-dependent embrittlement observed in stress-rupture tests and that produced by variations in heat-treatment,

(*d*) heat-treatment conditions yielding optimum combinations of smooth strength and notch properties for severe service applications.

The heat-treatment schedule used is given below:—

Steel	Austenitizing Temperature (½ hour)		Tempering Temperature		Tempering Time (hours)
	°F.	°C.	°F.	°C.	
17–22 A(S)	1650	900	1200	650	6
	1725*	940*	1200	650	6
	1850	1010	1200	650	6
	1975	1080	1200	650	6
	2075	1135	1200	650	6
	2075, 1725**	1135, 940**	1200	650	6
	1850, 1725**	1010, 940**	1200	650	6
	1725	940	Room		
	1725	940	800	425	6
	1725	940	1100	595	6
	1725	940	1100	595	20
	1725	940	1200	650	2
	1725	940	1200	650	6
	1725	940	1200	650	20
	1725	940	1300	705	0·5
	1725	940	1300	705	2
	1725	940	1300	705	6
	1725	940	1300	705	20
17–4 PH	1900	1035	1000	540	0·5
	1900	1035	1100	595	0·5

* Conventional heat-treatment ** Double normalizing

A single test temperature (1000°F. : 540°C.) was used for the chromium-molybdenum-vanadium steel; for the stainless steel the effect of temperature of test 300°–800°F. (150°–425°C.) was also studied.

The results reported show that notch-sensitivity and creep-embrittlement of both the steels studied are markedly influenced by variations in heat-treatment. In the chromium-molybdenum-vanadium steel normalizing at temperatures above 1725°F. (940°C.) intensifies notch-sensitivity and creep-embrittlement, and both are reduced by normalizing at lower temperatures, but with some loss in smooth strength. The creep-embrittlement and notch-sensitivity resulting from the high-temperature treatments are primarily associated with the carbide structure. Embrittled structures resulting from normalizing at high temperatures may be appreciably improved by re-normalizing at a lower temperature, without adverse effects on smooth strength.

For both steels, raising the tempering temperature reduces notch-sensitivity, with associated loss in smooth strength.

In the chromium-molybdenum-vanadium steel tempering at relatively high temperatures for short periods gives minimum notch-sensitivity and involves minimum reduction in smooth strength.

Carbide precipitation from ferrite can occur during tempering or during creep testing. If it occurs during testing its effects on the embrittlement and notch-sensitivity are considerably greater than if it takes place during tempering. Smooth strength is affected only if precipitation occurs during testing.

Vibrational Treatment of Austenitic Steel Castings during Solidification

S. HINCHLIFF and J. W. JONES: 'Application of Sub-sonic Vibrations during Solidification of Castings, with Particular Reference to a Material for Gas-Turbine Blades, H. R. Crown Max.'
Foundry Trade Jnl., 1955, vol. 99, Sept. 1, p. 251.
Abstract of Report No. 89 issued by the College of Aeronautics, Cranfield.

The authors report experiments in which sub-sonic vibrations, of varying frequency and amplitude, were applied during solidification of castings of austenitic steel within the following range of composition:—carbon 0·19–0·24, chromium 22·47–23·17, nickel 11·0–11·40, per cent.

The results of tests on material so treated indicate that the vibration has a generally beneficial effect on mechanical properties, and that grain size of the cast material is reduced by this means. Both frequency and amplitude are contributory factors: the former is the more important. The abstract gives a summary of conclusions drawn with regard to the influence of vibratory treatment on elongation, reduction of area, impact, proof-stress and tensile-strength values, and it is noted that the improved tensile strength so obtained is not accompanied by loss of ductility or impact-resistance, but that these properties also are enhanced. Particular attention is directed to the desirability of using a high pouring temperature (about 1550°C.), in order to ensure complete filling of moulds and thin cavities. Sub-sonic vibration applied to castings poured at that temperature gave a greater degree of improvement in properties than was obtained by identical treatment of castings poured at a lower temperature.

Descaling of High-Temperature Alloys

G. M. BRYAN: 'Improvement of Jet-Engine Descaling Procedure.' *U.S. Wright Air Development Centre Report* WADX-TR-52-100, 1952 (declassified 1954); 50 pp.

The report deals with the scaling characteristics of Inconel and the development of descaling procedures suitable for use on that alloy. X-ray analysis showed that the scale formed at high temperatures consists mainly of nickel oxide, with smaller amounts of chromium and iron oxides. Heavy scaling of Inconel appeared to be associated with precipitation, at the grain boundaries, of a phase which may be carbide and which may therefore explain the occurrence of intergranular corrosion of the alloy, after scaling, in acid solution. Neutral or alkaline solutions failed to remove scale from Inconel, but several acid solutions were developed by which the scale could be removed without serious attack on the underlying metal. Oxidizing pretreatments, especially with alkaline permanganate, promoted efficient removal of scale on subsequent pickling in acid solution. Inconel specimens which had been descaled in nitric acid/ferric chloride solution at 70°C. showed severe loss of mechanical strength, whereas treatment in the same solution at room temperature had no deleterious effect.

It is pointed out that the presence of scale on parts which are operating at high temperatures may be beneficial, in giving protection against further oxidation: the scaled surface is smooth, and is in some cases more regular than that of the descaled material.

Materials of Construction in Ore-Treating Plants

L. REEVE: 'Development of Chemical Treatment of Low-Grade Iron Ores at Appleby-Frodingham.'
Jnl. Iron and Steel Inst., 1955, vol. 181, Sept., pp. 26–40.

Reference is made to difficulties encountered in previous attempts at the Appleby-Frodingham plant to concentrate low-grade iron ores by purely physical methods, on which a report was made in 1949 (*ibid.*, vol. 159, pp. 275–80). The main paper comprises a detailed description of a cyclic chemical process which has since been developed for treating these ores. This involves the use of hydrogen-chloride gas or aqueous hydrochloric acid, in which the iron is distilled from the ores as pure ferric chloride, at 300°–350°C. The ferric chloride is then treated with steam, to produce pure ferric oxide, or with hydrogen to produce metallic iron: in both cases hydrogen-chloride gas is regenerated and can be re-used. Vanadium present in these ores can also be distilled, as a complex of vanadium chloride, and is subsequently recovered as V_2O_5. Chlorides remaining in the tailings can be regenerated by treatment

with steam mixed with air or with inert gases. Fluidizing techniques have been applied to these processes. Brief reference is made to the thermodynamics of the reactions involved, and to some of the chemical-engineering problems encountered in pilot-plant experiments.

A section of the paper is devoted to discussion of materials of construction used in the various sections of the plant required for the processes described. Search of the literature indicated that hydrogen-chloride gas is not an extremely corrosive agent so long as the gas is dry or at least above its dew-point. This conclusion was confirmed in a series of experiments in which the gas, with or without steam and/or air additions, was passed, for various periods, at temperatures up to 650°C., over mild steel, 18-8 chromium-nickel steel and 20-30 chromium-nickel steel (welded and unwelded). The laboratory results led to the conclusion that mild steel may safely be used for reactors and ducts at temperatures up to at least 350°C. Where direct contact with metal at temperatures over 400°C. and up to 650°C. is involved, stainless and heat-resisting steels of the types tested should be satisfactory. Experience in pilot-plant operation fully confirmed these findings: unprotected mild steel has been found adequate for hydrogen-chloride gas chloridizers at temperatures up to 350°C., and good service has been obtained with stainless steel or brick-lined mild-steel hydrolyzer shells working at temperatures up to 550°C. Internal pipes and distribution plates for use at these higher temperatures have been made from various grades of chromium-nickel stainless steel. The only corrosion encountered in such steel has been at positions in which the temperature has been allowed to fall below the dew-point temperatures of the particular gas mixture passing at these points. In such cases aqueous acid condensation has occurred.

High-Temperature Corrosion of Nickel and Other Metals by Nitric Oxide

M. FARBER, A. J. DARNELL and D. M. EHRENBERG: 'High-Temperature Corrosion Rates of Several Metals with Nitric Oxide.' *Jnl. Electrochemical Soc.*, 1955, vol. 102, Aug., pp. 446-53.

The authors report a study of corrosion over various temperature intervals within the range 800°-2000°K. (527°-1727°C.) of filaments of iron, tantalum, tungsten, molybdenum, nickel, copper, silver, Inconel (nickel-chromium-base alloy) and stainless steel (25-20 and 18-8 chromium-nickel types), in contact with nitric oxide.

The experimental method used involved measurement of the change of electrical resistance of a hot wire filament, 0·010-in. diameter, in the corrosive medium. Test equipment is illustrated in this paper: for fuller details of the technique, see *ibid.*, 1952, vol. 99, p. 427.

Observations made on the behaviour of nickel over the range 1100°-1650°K. (827°-1377°C.) are noted below:—

'Reaction rates increased steadily up to 1200°K. (927°C.). Above that temperature the rate starts to fall, to 1650°K. (1377°C.). The rate-determining step is presumed to be the formation of NiO, which forms a protective coating over the nickel. Two oxides of nickel are known to exist, Ni_2O_3 and NiO. Since at high temperatures the Ni_2O_3 decomposes to the lower oxide, it is believed that the rate of corrosion could probably be determined by the type of oxide formed, and to confirm this assumption X-ray diffraction patterns were obtained in both the high- and low-temperature regions. Oxide samples formed at 1100° and 1570°K. (827° and 1297°C.), at which temperatures the corrosion rates were identical, were analysed for crystal structure and type of oxide. Both showed only one type, NiO, but whereas the lower-temperature specimen consisted of large crystals, the high-temperature specimens showed small and compact crystalline structure.'

In the high-alloy group of materials, the corrosion rate of Inconel was negligible at all temperatures below the melting point, and no time-temperature curves could be made. The 25-20 stainless steel was almost as resistant as Inconel, corroding only slightly at 1550°K. (1277°C.) and above. The 18-8 steel corroded more readily.

A tabular summary of the initial corrosion rates of several metals at 1200° and 1450°K. (927° and 1177°C.) gave the following decreasing order of resistance:—

1200°K. (927°C.) silver, tungsten, nickel, iron, copper.

1450°K. (1177°C.) Inconel, 25-20 steel, molybdenum, nickel, tantalum, 18-8 steel, tungsten.

The results place these materials in the following general order of decreasing resistance to attack: Inconel, 25-20 chromium-nickel steel, molybdenum, nickel, tantalum, 18-8 stainless steel, tungsten, iron, copper.

Corrosion Tests in Water at Elevated Temperature

C. J. LANCASTER: 'Performance of Materials Tested in Water at High Temperature.'
U.S. Naval Engineering Experiment Station Report EES-4A(16)-966870, 1952; (declassified 1954); 12 pp.

Report of 30-day dynamic corrosion tests in which specimens of twelve materials were subjected to a static bending stress while rotating at a peripheral velocity of 11 f.p.s. in oxygenated water at 500°F. (260°C.). Weight losses and observations on the appearance of the specimens after test are recorded.

The materials exposed included 90-10 and 70-30 copper-nickel alloys, stainless iron and austenitic chromium-nickel steels, gold plating on stainless steel, brazing alloy on stainless steel, and an alloy containing nickel 25, chromium 17, molybdenum 5, per cent., balance iron. Weight changes in the copper-nickel alloys and in the brazing metal were so severe as to preclude their use in these conditions.

Chromium-Nickel-Manganese Steels

AMER. IRON AND STEEL INST.: 'New Stainless Steels containing 17%Cr-4%Ni-6%Mn and 18%Cr-5%Ni-8%Mn.'
Contributions to the Metallurgy of Steel, No. 47, May, 1955; 10 pp.

At the request of the Iron and Steel Division, Business and Defense Services Administration, U.S. Department of Commerce, the American Iron and Steel Institute has issued comparison data on steels of the chromium-nickel type (A.I.S.I. Type 301) and on lower-nickel, higher-manganese stainless steels.

A composition developed during the war period was approximately of the following type:—chromium 17, nickel 4, manganese 4, nitrogen 0·10-0·15, carbon 0·08-0·12, per cent. In the solution-annealed condition this steel had an austenitic structure, but under cold-working operations it transformed more readily than the 18-8 chromium-nickel types, and the instability of the austenite resulted in an exceptionally high rate of work-hardening, which introduced a hazard of cracking during forming. Later investigation has shown that increasing the manganese content to about 6 per cent. gives a steel the mechanical properties of which are closely similar to those of straight 17-7 chromium-nickel steel, and that the modified steels may safely be used as substitutes for 18-8 in some applications in which machinability and capacity for severe deformation are not essential. In cases where such characteristics are required an 18-5-8 chromium-nickel-manganese steel is more suitable. (See table below.)

Chemical Composition

Steel	C %	Si %	Mn %	P %	S %	Cr %	Ni %	N %
17-4-6	0·15 max.	1·00 max.	5·5/7·5	0·060 max.	0·030 max.	16·0/18·0	3·5/5·5	0·25 max.
18-5-8	0·15 max.	1·00 max.	7·5/10·0	0·060 max.	0·030 max.	17·0/19·0	4·0/6·0	0·25 max.

Tabular and graphical data contained in this report, which compare the properties of the new steels with 17-7 and 18-8 chromium-nickel types, are considered to justify the conclusion that mechanical and physical properties of the respective grades are broadly similar, that welding characteristics of the high-manganese steels are satisfactory, that their elevated-temperature properties compare favourably with those of 18-8 chromium-nickel grades, and that the resistance of the manganese-containing steels to atmospheric attack and to corrosion by some common corrodents is broadly similar to that of conventional 18-8 types. In confirmation of the atmospheric corrosion-resistance of the new steels, it is reported that The Budd Company has had satisfactory experience with their use in rail cars. That Company also finds that welding technique standard for the 17-7 chromium-nickel steels is applicable without modification to the manganese-containing types, and that the characteristics of the welds in the two materials are substantially identical. Forming practices used for Type 301 (17-7 chromium-nickel) have been successfully employed for the high-manganese steels.

Low-Nickel, High-Chromium Stainless Steel

R. A. LULA, W. G. RENSHAW and J. B. HILL: 'Low-Nickel Type 329 Steel Offers Good Corrosion-Resistance.' *Iron Age*, 1955, vol. 176, Sept. 8, pp. 74-6.

The writers urge the advantages, for certain applications, of steels containing chromium 25-30, nickel about 4 per cent., which structurally are closely similar to stainless steels of straight-chromium types. Reference is made, for example, to the successful use of this grade in Sweden, in equipment handling sulphite pulp. Typical microstructures of such steels, in various conditions, are illustrated, and the properties are discussed, indicating the behaviour of the steels as compared with fully austenitic higher-nickel compositions. Among the points to which attention is directed are the following:—

At about 2300°F. (1260°C.) the steels are completely ferritic, but austenite formed during cooling is stable at room temperature, and there is therefore no hardening by martensitic transformation. Carbides are always present, even in low-carbon heats of these steels, and grain-boundary precipitation will occur during rapid cooling from above 1800°F. (980°C.). As in other ferritic stainless steels, this change is associated with substantial lowering of resistance to corrosion: welded material is particularly susceptible to such deterioration.

The high-chromium, low-nickel steels are subject to both 885°F. (475°C.) embrittlement and to formation of sigma phase: if they are held for a sufficiently long time in the 1200°-1500°F. (650°-815°C.) range, a considerable proportion of sigma is formed, and although the accompanying hardening may in some circumstances be advantageous, such benefit is in many cases offset by simultaneous lowering of toughness. The mechanical characteristics of the steels are essentially those of the ferritic steels: average tensile properties of annealed material are given as u.t.s. 90,000-105,000 p.s.i. (40-47 tons per sq. in.), yield strength 70,000-85,000 p.s.i. (31-38 tons per sq. in.), and elongation 22-25, per cent. Welding of these lower-nickel steels can be easily accomplished, but post-weld full annealing is essential.

The major interest of the steels lies in their corrosion-resisting qualities. In chloride and in sulphuric-acid solutions they are superior, in the annealed condition,

to straight 18-8 chromium-nickel steel, and are its equal in resistance to nitric acid. By means of molybdenum additions, e.g., of the order of 1·5-2 per cent., good resistance to pitting attack is obtained, and behaviour in dilute sulphuric-acid solution at normal temperature is improved. Tests to date indicate that the high-chromium, low-nickel steels are susceptible to stress cracking in conditions which would cause such failure in the conventional higher-nickel types. The following figures are cited as typical of behaviour which may be expected in some common corrodents.

Corrosive Medium	Temperature	Corrosion Rate
Nitric acid, 65%	Boiling	0·00055 in./mth.
Glacial acetic acid	Boiling	0·000012 in./mth.
Phosphoric acid, 10%	Boiling	0·00086 in./mth.
Phosphoric acid, 85%	200°F. (95°C.)	0·0016 in./mth.
Sulphurous acid (conc. liquid)	150°F. (65°C.)	$1·7 \times 10^{-6}$ in./mth.
Sulphurous acid (vapours)	150°F. (65°C.)	$2·1 \times 10^{-6}$ in./mth. (no pitting)
Ferric chloride, 10%	Room	Pitted in 24 hours
Salt spray, 20%	Room	No rust or pitting in 8 mths.
15% HNO_3–3HF (by vol.)	Boiling	0·115 in./mth.

Stress-Corrosion Cracking in Hardenable Stainless Steels

F. K. BLOOM: 'Stress-Corrosion Cracking of Hardenable Stainless Steels.' *Corrosion*, 1955, vol. 11, Aug., pp. 351t-61t.

This detailed paper reports results of tests made by Armco Research Laboratories as an outcome of reports of cracking of high-chromium steels in various types of service. The investigation was concerned primarily with steels containing about 12-18 per cent. of chromium, with or without small percentages of nickel, but the following additional steels were included:—a modified 12 per cent. chromium steel containing molybdenum and tungsten, a precipitation-hardening chromium-nickel-copper steel (17-4 PH), a precipitation-hardening chromium-nickel steel containing aluminium (17-7 PH), a precipitation-hardening grade of 17-10 chromium-nickel steel, two types of nominal '18-10' chromium-nickel steel (one rendered magnetic by severe cold work, the other non-magnetic), two low-alloy steels (chromium-molybdenum and nickel-chromium-molybdenum grades), the low-manganese steel known in the oil industry as N80, and the 9 per cent. nickel steel which has also found application in the oil industry.

The environments selected to form the test conditions were chosen in the light of earlier studies of stress-corrosion which had been reported in the literature. Laboratory tests were made on most of the materials in a solution of distilled water containing ½ per cent. of acetic acid (HAc), periodically saturated with hydrogen sulphide. A modification of this solution, containing 6 per cent. of sodium chloride, was also used. (These media were originated by Shell Development Company, to simulate conditions found to cause cracking in sour gas condensate wells.) Two further sets of tests consisted in exposure to 20 per cent. salt-spray 'fog', and immersion in an aqueous 6 per cent. solution of sodium chloride. In addition, specimens of most of the materials were exposed in two types of atmosphere: (1) relatively severe marine conditions, and (2) a severe industrial atmosphere containing fumes from manufacturing plants and chemical laboratories.

Two methods of test were employed. In most cases a simple horseshoe-shaped sample was used, formed from annealed wire ¼-in. in diameter, heat-treated, descaled without contacting acids, and polished. Such samples were loaded to a nominal outer fibre stress of 100,000 p.s.i. (44·5 tons per sq. in.) by pulling the legs of the horseshoe together with an austenitic chromium-nickel steel wire clamp. The second test method provided a precise means of applying a known tension stress to the samples. Special machines were constructed, designed on the lines of those used for high-temperature creep and rupture testing. Tensile-type specimens were dead-weight loaded by levers, and the specimens were surrounded by containers holding the corrosive media. (Specimens, method of testing, and typical fractured specimens are illustrated for both series.)

Results of the individual tests are reported in extenso, and the reaction of the respective materials in the various conditions is critically considered in relation to composition, condition of heat-treatment, and other variables.

It is concluded that martensitic hardenable steels are susceptible to cracking under the combined influence of corrosion and tension stress, either externally applied or present as internal residual

stresses resulting from quenching. Such cracking is apparently common to all high-strength hardened materials. The likelihood of cracking is dependent on the type of corrosive medium with which the material is in contact, the hardness of the steel or alloy, the heat-treatment to which it has been subjected, the level of applied stress, and the type of material.

The media presenting the most severe conditions appear to be those which promote hydrogen embrittlement, such as acid-sulphate solutions and solutions containing chlorides which promote corrosion pitting. In the acetic acid-hydrogen sulphide test cracking occurred at hardness levels as low as Rockwell C 24; in severe marine atmospheres at C 40 and above, and in severe industrial atmospheres at C 45. Salt fog and aqueous salt solutions caused cracking at C 45 and above.

The precipitation-hardening steels 17-4 PH and 17-7 PH, if aged to their maximum hardness, were susceptible to stress-corrosion cracking, but if overaged were among the most resistant of the materials tested. For exposure to severe industrial or marine atmospheres under high stresses the overageing temperature usually recommended (1050°F.: 565°C.) provides satisfactory immunity to cracking in 17-7 PH: for similar exposure 17-4 PH should be overaged at 950°-1000°F. (510°-540°C.). For service in extremely severe conditions, such as are represented by the acid-hydrogen sulphide test, both steels are likely to give good resistance when overaged at 1150°F. (620°C.), to a hardness of about Rockwell C 30. When tempered to the same hardness level, the chromium-molybdenum-tungsten steel also gave good results in similar media.

Severely cold-worked 18-10-type chromium-nickel steels cracked only in the acid-hydrogen sulphide medium containing sodium chloride, and the same result was recorded for the precipitation-hardened 17-10 chromium-nickel steel hardened to Rockwell C 30. Cracks in these steels had a typical transgranular branched appearance and were different from those found in the martensitic steels. No difference in resistance to cracking was noted between the magnetic and non-magnetic specimens of the '18-10' composition.

Internal stresses generated in quench hardening can be sufficiently high to promote stress-corrosion cracking in the hardenable grades of steel. Some indication was found that interrupted quenching (martempering) is helpful in inhibiting susceptibility. In oil-quenched low-carbon 12 per cent. chromium steel tempering at 1000°-1050°F. (540°-565°C.) reduced internal stresses to a level sufficiently low to prevent cracking in the severest media used.

It is considered likely that stress-corrosion cracking in hardened stainless steels is accelerated by hydrogen generated by corrosion and absorbed in the steel. The cracks start in many cases from pits developed on the surface of the specimen, and are primarily, but not exclusively, transgranular. They are similar in appearance to cracks produced by hydrogen cathodically introduced.

Austenitic Chromium-Nickel Steel in Bone Surgery

C. A. ZAPFFE: 'Human Body Fluids Affect Stainless Steel.' *Metal Progress*, 1955, vol. 68, July, pp. 95–8.

Research carried out prior to and during the war period led to the conclusion that chromium-nickel steel, of the straight 18–8 or molybdenum-containing type, was the most suitable material for use in bone fixation. The extensive tests made had included study of the behaviour of these steels and of other materials in physiological salt solutions and serum, also numerous *in vivo* experiments. In 1943 austenitic steels of these grades were recommended for standardization in American Service hospitals.

Subsequent research has revealed that such steels are more susceptible than had previously been believed to 'mechanochemical' or stress cracking, i.e., cracking resulting from the joint action of mechanical stress and chemical attack. The phenomenon was dealt with in considerable detail in a symposium organized in 1944, by the American Society for Testing Materials and the American Institute of Mining and Metallurgical Engineers, but the data then presented related mainly to cracking in solutions of somewhat special composition, used in strong concentration. Since that date it has been increasingly realized that susceptibility to such deterioration is more general than had been anticipated, and the present writer cites supporting evidence which is relevant to the use of the austenitic steels in surgery. The first relates to a Collison plate, used in humerus fixation. The nature of the cracking is illustrated, and a record of examination of the failure (in which the possibility of other causes was also fully investigated) indicates that it was an example of stress cracking, with the body fluids acting as the corrodent. Supporting evidence on stress cracking, although in a different application, is reported, comprising failure of a specimen of stainless steel exposed to the liquor of an ammonium bisulphite digester under stressed conditions. A third example which is of interest in this connexion concerns the back cover of a watch-case. The back was attached to the case by an internal thread, requiring a special spanner wrench seating in eight depressions placed octahedrally round the circumference. The depressions had served as sites of residual internal stress, and human perspiration had acted as corrodent.

Corrosion Research in the U.S.A.

F. L. LAQUE: 'Attention to Corrosion in the U.S.A.' *Chemistry and Industry*, 1955, Aug. 13, pp. 1016–25. Reprint of Spring Lecture delivered to the Corrosion Group, Society of Chemical Industry, London, March 30, 1955.

The lecture provides a comprehensive review of corrosion research and testing in progress and immediately projected in the U.S.A. An account is given of the terms of reference and the current activities of technical societies, Governmental agencies and some industrial organizations engaged in major corrosion projects: the survey also includes information on educational facilities available in the

U.S.A. for those wishing to specialize in corrosion aspects of chemical engineering and related subjects, and on research in progress in the universities. In spite of the volume of investigation now under way, the position is considered inadequate, in view of the practical importance of corrosion as an economic factor, and there are plans for formation of a Corrosion Research Council, whose object will be to inaugurate a programme of fundamental research on a scale not yet realized. To this end industrial firms in the U.S.A. are being asked to make a major contribution.

Corrosion Testing under Atmospheric and Marine Conditions

M. G. FONTANA: 'Corrosion. INCO's Corrosion Testing Stations in North Carolina.'
Industrial and Engineering Chemistry, 1955, vol. 47, July, pp. 69A–71A; Aug., pp. 87A–8A.

A detailed description of the two corrosion-test stations maintained by The International Nickel Company in North Carolina, one at Kure Beach (where atmospheric exposure tests are made), the other at Harbor Island (where sea-water testing is carried out).

At the Kure Beach site tests include exposure at various distances from the sea, and the effect of various degrees of shelter is also observed. At Harbor Island sea-water attack is studied in numerous conditions simulating all types of marine service, to provide data which can serve in selection of material suitable for piling, condenser tubing, pumps, and other parts operating in sea water. Laboratory-scale tests also play an important part in the test programmes: some of the methods used are described in these notes.

Intergranular Corrosion Test for Austenitic Stainless Steels

AMER. SOC. TESTING MATERIALS: 'Tentative Recommended Practice for Conducting Acidified Copper Sulphate Test for Intergranular Attack in Austenitic Stainless Steel.'
Contained in Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys.' *A.S.T.M. Preprint* 7, June, 1955, pp. 5–8.

This schedule, put forward for adoption as tentative at the Society's 1955 meeting, covers procedure for conducting one of the standard tests used to measure susceptibility of austenitic stainless steels to intergranular attack. Detailed recommendations are made on apparatus required, composition of test solution, sensitizing treatment to be applied to the steels, immersion exposure conditions, bend-test technique, and mode of evaluating the results. It is emphasized that the presence or absence of attack in this test is not necessarily a criterion of the performance of the material in other corrosive environments.

See also—

'Proposed Revision of Tentative Recommended Practice for Boiling Nitric-Acid Test for Corrosion-Resisting Steels', A 262-52T. *ibid.*, pp. 9–13.

After extensive examination of the potential usefulness of the oxalic-acid etch test, the incorporation of this procedure as a 'screening' method is recommended, for use in conjunction with the boiling nitric-acid (Huey) test on stainless steels. (Reference is made in the specification to original descriptions of electrolytic oxalic-acid etching which have been published during the past few years: for summaries see *Nickel Bulletin*, 1954, vol. 27, Nos. 3 and 7–8, pp. 52 and 151.)

It is proposed (1) that materials which qualify as satisfactory by the oxalic-acid test need not be subjected to the boiling nitric-acid test, and (2) that materials which do not pass the etch test should be subjected to the boiling nitric-acid test as a final basis for acceptance or rejection.

The oxalic-acid test is applicable only to certain types of austenitic (wrought and cast) steels, which are listed in the specification. Test technique is detailed, and 'acceptable' and 'unacceptable' structures, as revealed by oxalic-acid etching, are illustrated in the original.

Huey Boiling Nitric-Acid Test: Influence of Contamination of Solution

R. J. BENDURE: 'Acid Contamination as a Source of Error in Boiling Nitric-Acid Test for Corrosion-Resistant Steels.' *A.S.T.M.Bull.*, 1955, No. 207, July, pp. 76–7; T.P. 149.

The letterpress of this note is substantially similar to that contained in *Corrosion*, 1954, vol. 10, Mar., *N.A.C.E. News*, p. 1: see abstract in *Nickel Bulletin*, 1954, vol. 27, No. 5, p. 96. In the A.S.T.M. Bulletin tabular data are presented demonstrating the erratic results due to contamination of nitric-acid test solutions by fluorides.

Molybdenum-containing Materials in the Chemical-Engineering Industries

The August 1955 issue of *Industrial and Engineering Chemistry* includes a survey of industrial applications of molybdenum, as presented in a symposium before the American Chemical Society. Individual contributions deal, respectively, with the use of molybdenum in catalysts, in inorganic pigments, in organic pigments, in metal coatings, and in alloys for the chemical-process industries. The scope of the last-named paper is shown below.

J. Z. BRIGGS: 'Alloys for the Chemical Process Industries', pp. 1513–16.

The use of molybdenum in corrosion-resisting materials is reviewed on the basis of its employment:

(1) *as an addition agent in austenitic chromium-nickel steels*, where in some cases it enhances general corrosion-resistance, and in others confers a degree of resistance to attack by a particular corrosive which is not obtainable in molybdenum-free steels. Typical cases in which molybdenum additions are beneficial include materials for use in organic syntheses and processing, (particularly those involving organic acids of relatively low molecular weight) and in some inorganic processes for which molybdenum-free grades give inadequate resistance, (e.g., in handling

certain concentrations of phosphoric, sulphuric, and chromic acids, and mixtures of nitric and hydrofluoric acids). Molybdenum is also used to enhance pitting-resistance of austenitic steels, and, in strictly controlled proportions, to increase high-temperature strength of such steels.

(2) *as an addition, in association with copper, to austenitic chromium-nickel steels*, especially those employed in sulphuric-acid service. Particular attention is directed to steels containing chromium 20, nickel 30, molybdenum 2·5, copper 3·5, per cent.

(3) *in high-silicon silicon-iron alloys*, where the presence of molybdenum enhances resistance to attack by hot hydrochloric-acid and chloride solutions.

(4) *in nickel-molybdenum-base alloys*, which have outstandingly high resistance to attack by hydrochloric-acid solutions. As typical of the behaviour of such materials, it is stated that a nickel-molybdenum alloy containing about 28 per cent. of molybdenum has high resistance to hydrochloric acid of all concentrations and at all temperatures. With lower molybdenum (about 21 per cent.) and an iron content of about 19 per cent. the alloys are resistant to all concentrations, but only to a temperature of 160°F. (70°C.). Modification of the nickel-molybdenum-base alloys by additions of chromium and tungsten is also advantageous for use under certain conditions.

(5) *as the pure metal*, to give resistance to specific media such as hydrofluoric acid, some phosphoric-acid solutions, and molten sodium or sodium vapour.

(A further paper, on the rôle of molybdenum in agriculture, was published in the August 1955 issue of *Agricultural and Food Chemistry*.)

Metallic Materials for Nuclear-Fuelled Power Plants

See abstract on p. 176.

Determination of Cobalt in Austenitic Stainless Steels and Nickel-base Alloys

L. P. PEPKOWITZ and J. L. MARLEY: 'Rapid Determination of Cobalt in Alloy Steels by the Tetraphenylarsonium Method.' *Analytical Chemistry*, 1955, vol. 27, Aug., pp. 1330–1.

Potratz and co-workers have recently described a colorimetric method for determination of cobalt in steel, using the tetraphenylarsonium cobaltothiocyanate complex (*ibid.*, 1949, vol. 21, p. 1276; 1951, vol. 23, p. 1680). The procedure is rapid, and is stated to give results as accurate as those obtained by the reference (electrolytic) method. In the present paper the authors describe a modified form of the Potratz method, adapted for the determination of cobalt in stainless steels of the chromium-nickel types, in nickel-chromium-iron alloys, and in various other high-alloy types of material.

Cladding of Metals by Vacuum Brazing

See abstract on p. 175.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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